

# The Journal *of the* Society of Dyers and Colourists

Volume 77



Number 6

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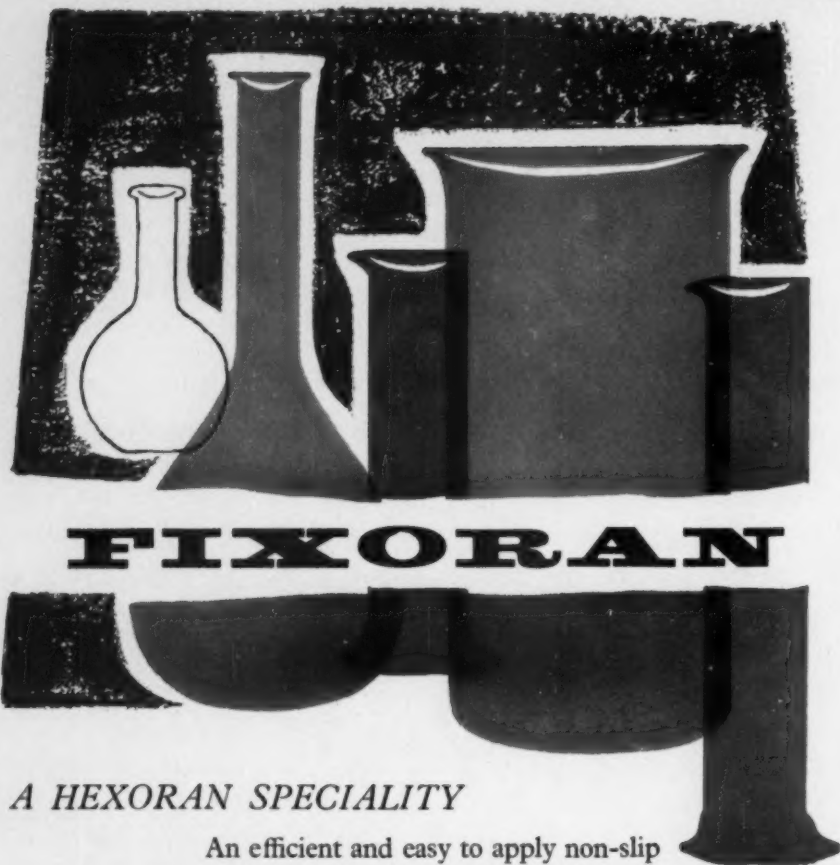
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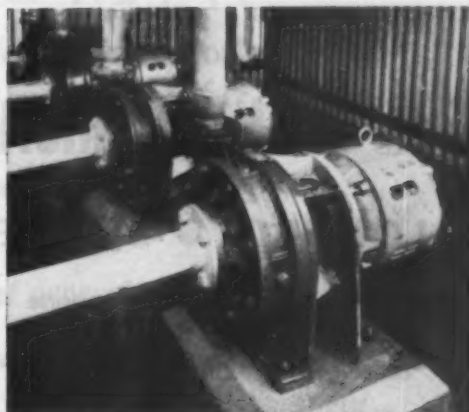
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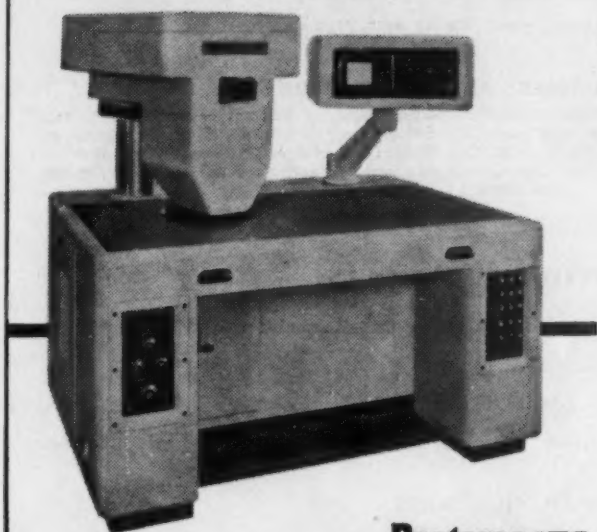
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(Abstracts section only—see January issue, page 2)

## NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1-8 of the January 1961 and pages 389-396 of the July 1960 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

## Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

### LECTURES

Some Difficulties in the Coloration of Plastics

*J. M. J. Estevez*

### COMMUNICATIONS

Setting of Animal Fibres in Solutions of Thiourea Dioxide

*L. S. Bajpai and C. S. Whetwell*

Interaction between some Acid Wool Dyes and Nonyl  
Phenol-Ethylene Oxide Condensates

*B. R. Craven and A. Datyner*

Effect of Sulphuric Acid on Wool. V— Investigation  
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*R. L. Elliott, R. S. Asquith, and B. J. Jordan*

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# THE JOURNAL OF THE Society of Dyers and Colourists

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Volume 77 Number 6

JUNE 1961

Issued Monthly

## Proceedings of the Society

### Recent Developments in the Dyeing of Acrylic Fibres

B. KRAMRISCH

*Meetings of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 20th September 1960, Mr. J. G. B. McCallum in the chair; of the Manchester Junior Branch held at the Manchester College of Science and Technology on 17th November 1960, Mr. A. S. Fern in the chair; and of the West Riding Section held at the Victoria Hotel, Bradford, on 23rd February 1961, Mr. J. Rankin in the chair*

Recent developments and problems encountered in the application of disperse, cationic and anionic dyes to various types of acrylic copolymer fibres are discussed. High-temperature dyeing with 1:2 metal-complex dyes is considered, with reference to the influence of application conditions. Dyeing of unions of acrylic fibres with both natural and man-made fibres by several one-bath and two-bath methods is described. Finally, some results are given on the dyeing of nitrile alloy fibre (Zefran), comparing the fastness of dyeings on this fibre with the fastness obtained on cotton and on wool.

#### Introduction

The growing importance of acrylic fibres is indicated by a recent comparison<sup>1</sup> of the projected production in Great Britain of polyacrylonitrile, polyamide, and polyester fibres with similar figures for polyacrylonitrile fibres in the U.S.A. The comparative quantities (production of Acrilan in Great Britain = 1) are—(G.B.) Acrilan 1, Courtelle 3.2, nylon 9, Terylene 5; (U.S.A.) Acrilan 4.2, Creslan 2.7, and Orlon 20.

The principal textile uses of acrylic fibres are in knitted outerwear and (in unions with wool or spun viscose rayon) suitings and dress materials. Interest has also spread to blankets, carpets, and scatter rugs. One type of acrylic fibre, Dynel, has found application in imitation fur coats and in the hatting industry.

Acrylic fibres can be classified into four different chemical groups. In this paper, reference will be made mainly to those acrylic fibres which are either manufactured in or imported into Great Britain, but some attention will also be paid to other American-made acrylic fibres of general technical interest.

*A Polymers containing at least 85% acrylonitrile*  
Orlon 42 E. I. Du Pont de Nemours & Co. Inc.  
Wilmington, Delaware, U.S.A.

*Courtelle* Courtaulds Ltd., Coventry, England.

*Acrilan 16 (or C3)* Chemstrand Corp., Decatur, Alabama, U.S.A.

#### B Basic Acrylic Copolymers

*Acrilan* Chemstrand Corp., Decatur, Alabama, U.S.A.\* (Regular Acrilan or CI).

*Creslan* American Cyanamid Co., Fibres Division, New York, U.S.A.

#### C Polymers of Acrylonitrile with Vinyl or Vinylidene Compounds

*Dynel* Union Carbide Chemicals Co., Division of Union Carbide Corp., New York, U.S.A.

*Verel* Tennessee Eastman Corp., Kingsport, Tennessee, U.S.A.

#### D Nitrile "Alloy"

*Zefran* Dow Chemical Co., Midland, Michigan, U.S.A.

As detailed general references to the dyeing of most of these fibres have already been published<sup>2</sup>, this paper will deal mainly with more recent developments and special points of interest or difficulties which have been encountered. Consideration will also be given to new developments in the production of solid dyeings and two-colour effects on unions of acrylic fibres with natural and man-made fibres.

#### Disperse Dyes

In general, disperse dyes provide dyeings sufficiently fast to light and to washing for ladies' knitted outerwear and dress goods. Careful selection is, however, necessary if the dyed

\* Acrilan is now manufactured at the Chemstrand factory in Coleraine, Northern Ireland.

materials have to withstand steaming treatments, e.g. permanent pleating or Hoffman pressing.

One defect of many disperse dyes is that they do not build up well to full depths on acrylic fibres; the behaviour depends on the individual dye and the type of fibre. Examples illustrating the behaviour on acrylic fibres and on nylon are—

**Cibacet Yellow 2RG (C.I. Disperse Yellow 36)**

Has little affinity for Courtelle and Orlon. Affinity for Acrilan and Creslan is moderate, but appreciably less than affinity for nylon, on which it builds up progressively to full depths.

**Cibacet Brilliant Scarlet RG (C.I. Disperse Red 12)**

Has very little affinity for Orlon and only slightly higher affinity for Courtelle. Has good affinity for both Acrilan and Creslan, on which it builds up almost as well as on nylon.

**Cibacet Sapphire Blue G (C.I. Disperse Blue 1)**

Builds up well on all types of acrylic fibres, almost as well as on nylon.

Courtaulds now recommend that dyeing of Courtelle with disperse dyes be carried out under slightly acidic conditions to achieve maximum brightness; at the same time a non-ionic agent should be present in the dyebath. To meet these requirements dyeing can be carried out with 1 g/l. sodium dihydrogen phosphate or 0.5 ml/l. acetic acid (80%), together with 1 g/l. Emulsifier OC (non-ionic dispersing agent) for 1½ h at the boil.

One difficulty which may be encountered with certain disperse dyes is the tendency to form dark crystals on slow cooling after dyeing; this is aggravated when washing-off after dyeing is not practised. As a result the final dyeings are apparently unlevel and are difficult to correct. Many pink disperse dyes are very prone to this defect. Furthermore, if these dyes are applied under alkaline conditions, uneven bluish patches may be formed on the material. These can subsequently be removed by treating the material under acid conditions, e.g. with 0.5 ml/l. glacial acetic acid and 1 g/l. Emulsifier OC for 1 h at the boil. If the pH of the dyebath is too high, the dye may even be destroyed.

Other disperse dyes are liable to be affected when applied at high pH values, but the effect varies with the individual dye.

### Basic Dyes

Basic and modified basic dyes have proved very useful for medium to full colours on Orlon 42, Courtelle, regular Acrilan, and Acrilan 16. On regular Acrilan basic dyes are best applied with 3% urea and a non-ionic product, e.g. Emulsifier OC, and on Orlon 42, Courtelle, and Acrilan 16 from an acetic acid dyebath. Non-ionic products and retarders may also be employed, but careful control of temperature is essential for achieving level results.

It is claimed that basic dyes may also be used for pale colours, mainly on loose stock and sliver.

Basic dyes give a wide range of colours on both Dynel and Verel. Both ordinary and modified basic (Deorlene) dyes give a wide range of colours and depths on Verel on application in absence of a

carrier, viz. with 3% urea and 2% Emulsifier OC for 1½ h at the boil. Some increase in depth is obtained with the Deorlene blacks by adding 5 ml/l. Invalon PR (anionic carrier for synthetic fibres) as carrier—the light fastness is unaffected.

Ordinary and modified basic dyes can be applied to Dynel also by the urea technique. In most cases, however, distinct improvement in yield is achieved by including 3–5 ml/l. Invalon PR—this is particularly striking with the Deorlene blacks, which have very little affinity in absence of a carrier, giving only weak, dull greens. In general a good standard of wash fastness is achieved whether dyeing takes place in presence or in absence of a carrier. The fastness to steaming varies with the individual dye.

Dyeings of ordinary basic dyes have low to moderate light fastness irrespective of the method of application; they are somewhat inferior to the modified basic dyes. In general the light fastness of a particular dyeing on Dynel is similar to that on Verel.

Increase in depth of colour is achieved by heat relustring, e.g. at 115°C. The final depth obtained on Dynel or Verel tends to vary significantly with drying conditions, particularly with drying temperature; the behaviour is similar to that of Acrilan dyed with anionic, e.g. acid, dyes.

The thermoplastic properties of Dynel and Verel must be taken into consideration during dyeing and finishing, and are of particular importance when carriers are used. Variations in hue during processing may be difficult to control in bulk and make reproducibility difficult.

### Acid Dyes

Although acid dyes permit the production of a wide range of colours on regular Acrilan, two points merit special attention.

#### LIGHT FASTNESS

The light fastness of dyeings of acid dyes on Acrilan varies with the conditions of application<sup>2</sup>. Furthermore, individual dyes may yield dyeings on Acrilan and wool of markedly different light fastness. This effect is illustrated in Table I.

TABLE I  
Fastness to Light of Acid Dyes on Acrilan and on Wool

Dye	Colour Index No. C.I. Acid	Light fastness* (daylight)	
		Acrilan	Wool
Kiton Fast Yellow 2GL	Yellow 17	6	7
Kiton Crimson 2R	Red 14	3	3
Benzyl Red MG	Red 97	1–2	4
Benzyl Fast Red 3G	Red 104	3	5
Alizarine Fast Blue BE	Blue 192	4–5	5–6
Alizarine Fast Green 2GW	Green 40	5	6
Alizarine Fast Grey G	Black 50	4–5	4–5

\* Acrilan was dyed from a bath containing 6% sulphuric acid, and wool by conventional methods, both for 1 h at the boil. All dyeings were at standard depths.

A further difficulty is that the light fastness of mixture dyeings on Acrilan is not necessarily as high as that of self-colours, in contrast to the behaviour on wool. For example, Kiton Fast

Yellow 2GL tends to give dyeings of lower light-fastness on Acrilan in mixture dyeings with other acid dyes and is best replaced by a 1:2 metal-complex dye, e.g. Cibalan Yellow GRL (C.I. Acid Yellow 116), which is free from this defect and is compatible with acid dyes at the low pH at which dyeing takes place.

#### DRYING TEMPERATURE

Variations in drying temperature have a marked influence on the final depth produced, which increases with increase in drying temperature. Furthermore, if dyed Acrilan is wetted out and air dried, the effect is reversed.

This difficulty can give rise to trouble in bulk processing, e.g. when drying Acrilan carpet yarn, and may arise during finishing when the backs of carpets are steamed. This effect is encountered to varying degrees with other anionic dyes and to a lesser extent with disperse dyes on Acrilan.

#### Reactive Dyes

Reactive dyes will dye Acrilan under acid conditions, but in general the light fastness is appreciably lower than that obtained with the same dyes on wool or cellulosic fibres. Reactive dyes will not dye Orlon, Courtele, and Acrilan 16 under normal conditions. Reference has, however, been made by Dow Corning<sup>4</sup> to a process for dyeing Orlon with reactive dyes. This consists in pre-treating Orlon with a water-dispersible silicone, padding with a solution of a reactive dye, drying at 125°C to fix the dye, and rinsing in a hot soap solution to remove excess dye before finally scouring.

#### High-temperature Dyeing of Acrylic Fibres

Dyeing at temperatures above 100°C was first used commercially for the application of disperse dyes to polyester fibres; it is also used to some extent on polyamide fibres. Sometimes it assists in promoting levelling and in reducing dyeing time; in addition, wet-fastness properties, e.g. fastness to washing, tend to improve progressively with increase in application temperature.

This behaviour is illustrated by Fig. 1, which relates the washing fastness (S.D.C. Mechanical

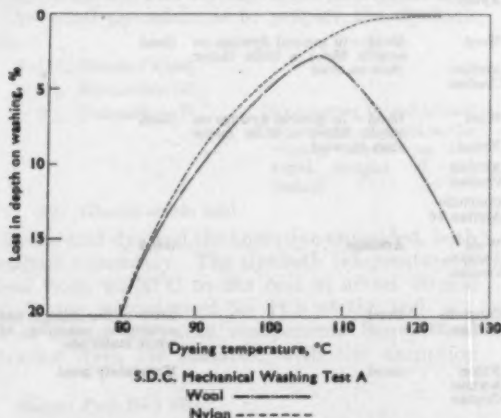


FIG. 1—Effect of dyeing temperature on fastness to washing of dyeings of 1% Cibalan Red 2GL on wool and on nylon

Washing Test A) of dyeings on nylon of a 1:2 metal-complex dye, Cibalan Red 2GL (C.I. Acid Red 211), to temperature of application. It indicates that maximum washing fastness is achieved by dyeing at high temperatures. Comparison with the behaviour of dyeings on wool shows that the washing fastness on wool also improves progressively with increase in application temperature to a maximum at 105–107°C. At higher temperatures the washing fastness falls rapidly owing to degradation of the wool.

High-temperature dyeing of acrylic fibres has been practised commercially using metal-complex dyes, particularly the 1:2 complexes<sup>5</sup>. Dyeing is best carried out at 120–130°C in presence of electrolytes, e.g. common salt; in some instances a small amount of acetic acid is needed to facilitate exhaustion of the dyebath. This method is particularly valuable for Courtele, Orlon 42, and Acrilan 16, as these fibres are dyed to only a slight extent unless temperatures above the boil are used.

A wide range of colours is obtainable with very high fastness to light and to wet treatments, e.g. washing, alkaline milling, and steaming, comparable to those obtainable on wool with the faster wool dyes.

The influence of dyeing temperature on the uptake of a 1:2 metal-complex dye, Cibalan Bordeaux RL (C.I. Acid Violet 71), by four types of acrylic fibre—regular Acrilan, Creslan, Orlon, and Courtele—and by nylon is illustrated in Fig. 2.

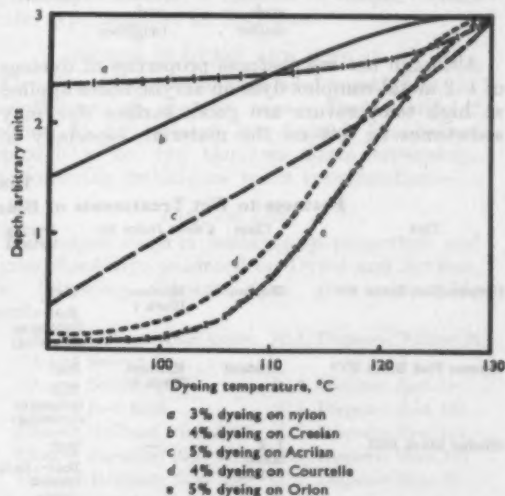


FIG. 2—Effect of dyeing temperature on depth of Cibalan Bordeaux RL on acrylic fibres and on nylon

Variations in the amount of electrolyte and in liquor ratio have a significant effect on the uptake of 1:2 metal complexes, as illustrated in Fig. 3.

The colour obtained varies with the substrate, as indicated in Table II, which compares the colours obtained with several 1:2 metal-complex dyes applied to acrylic fibres at 130°C by the method of BP 777,534<sup>5</sup> with those obtained on wool dyed at the boil. The colour obtained on nylon dyed at the boil is also included.

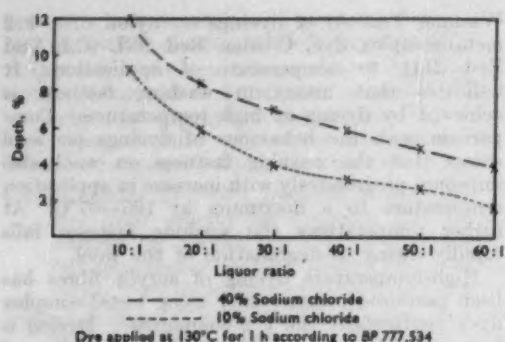


FIG. 3—Effect of liquor ratio and salt concentration on depth obtained with Cibalan Black BGL on Courtele

TABLE II  
Cibalan Dyes on Various Substrates—Comparison of Colour obtained with that on Wool

Dye Substrate	Cibalan Bordeaux EL	Cibalan Brown VRL	Cibalan Corinth BL	Cibalan Blue 3GL (C.I. Acid Blue 171)
Nylon	Similar	Much redder	Similar	Redder
Courtele	Similar	Duller and much redder	Similar	Redder
Orlon	Similar	Duller and much redder	Much redder and brighter	Redder
Acrlan	Similar	Redder and duller	Redder and brighter	Redder

Although the wet-fastness properties of dyeings of 1:2 metal-complex dyes on acrylic fibres applied at high temperature are good, surface dye may sometimes be left on the material, especially in

deep colours. This can be removed by after-treatment with a solution of 0.5 g/l. of soda ash and 1 ml/l. of a non-ionic detergent, e.g. Ultravon JU for 30 min at 45–50°C. As a result the treated dyeings show the maximum fastness.

Addition of a small amount (1%) of glacial acetic acid facilitates the exhaustion of several 1:2 metal-complex (e.g. Cibalan) dyes on Courtele. This is true also of most 1:2 metal-complex dyes on Acrlan. The treatment also effects an improvement in certain wet-fastness properties.

#### Production of Blacks on Acrylic Fibres

Blacks and charcoal greys account for a substantial proportion of dyed wool, particularly slubbing and loose wool. This is true also of mélange-printed wool. Blacks are of equal importance on all types of synthetic fibres and, where dyed wool and synthetic fibre are to be blended for the production of yarns and fabrics, the two materials should have similar fastness. For dyed wool, apart from fastness to light, fastness to certain wet treatments, e.g. washing, alkaline milling and steaming, and to perspiration, is of importance. Alkaline milling is required for coatings and steaming for materials which are to be permanently pleated or Hoffman pressed.

To study the wet fastness of blacks produced on wool and on acrylic fibres, typical chrome and metal-complex (1:1 and 1:2) dyes were applied in full depths on wool and, where possible, on acrylic fibres, if necessary by a modified technique. In addition, blacks were also produced with modified basic dyes and by the modified azoic process. Similar dyeings were prepared on nylon, where applicable, for general comparisons. These dyeings were then compared for fastness to alkaline milling and to steaming. The results, which are summarised in Table III, indicate that there are

TABLE III  
Fastness to Wet Treatments of Blacks on Acrylic Fibres, Wool, and Nylon

Dye	Class	Colour Index No. C.I.	Dyeing Method	Substrate	Fastness to Alkaline Milling (S.D.C. 3rd Report 2nd Edn)	Steaming (15 lb/in <sup>2</sup> pressure, 15 min)
Chrome Fast Black A*	Mordant	Mordant Black 1	Boil Boil (intensive chroming)	Wool Nylon	Good	Good
Chrome Fast Black PV*	Mordant	Mordant Black 9	Boil Boil (intensive chroming)	Wool Acrlan Creslan	Good—in general dyeings on acrylic fibres a little faster than on wool	Good
Cibalan Black BGL	1:2 Metal complex	—	Boil Boil—back tanned 120°C† 130°C†	Wool Nylon Acrlan Creslan Courtele Acrlan 16	Good—in general dyeings on acrylic fibres a little faster than on wool	Good
Combination of Neolan Black WA extra and Neolan Black 2G	1:1 Metal complex	Acid Black 52 Acid Black 54	Boil	Wool Acrlan Creslan	Average	Good
Decliene Black K4059	Modified basic	—	Boil	Courtele Acrlan 16	Good	Little colour change, but noticeable staining of white materials
Combination of Cibaset Diazo Black HD and Cibasaphthol ETO	Azoic	Azoic Coupling Component 18	Modified azoic process	Nylon Acrlan Creslan	Good	Moderately good

\* Applied by afterchrome process.

† Applied by method of BP 777,534.

several methods of producing blacks on acrylic fibres comparable in wet fastness with blacks on wool or nylon.

### Dyeing Unions Containing Acrylic Fibres

Information has already been published<sup>2</sup> on methods of obtaining reserve, solid and two-colour effects on unions of acrylic fibres with cellulosic fibres, wool, silk, nylon, and cellulose triacetate. Consideration will now be given to some recent developments in this field.

#### UNIONS OF COURTELLE, ORLON 42, AND ACRILAN 16 WITH WOOL (ONE-BATH PROCESS)

Earlier recommendations<sup>2</sup> were based on the use of selected wool dyes together with basic (Deorlene) dyes applied from an acetic acid dyebath containing Emulsifier OC (non-ionic agent) as anti-precipitant. Kiton Fast Yellow 2GRL (C.I. Acid Yellow 29), Kiton Fast Red 3GP (C.I. Acid Red 57), and Kiton Fast Blue 4GL (C.I. Acid Blue 23) were suggested for pale to medium depths. For medium to full depths, the more level-dyeing milling dyes, e.g. Alizarine Fast Blue BE (C.I. Acid Blue 129), were suggested and, in addition, reactive (Cibacron) and the allied Cibacrolan dyes for individual bright deep colours.

The drawbacks of these recommendations lay in the inadequate wet-fastness properties of Kiton Fast dyes in deeper colours and also in the limited number of milling acid dyes which are sufficiently level dyeing in presence of acetic acid. Furthermore, the Cibacron and Cibacrolan dyes were not sufficiently level-dyeing when applied by the recommended method, and they provided only a limited range of colours.

This situation has since been changed by the introduction of Univadine W (non-ionic levelling agent)<sup>6</sup>, which permits wool to be dyed level with most milling dyes in the presence of acetic acid, which is required for the application of the basic dye to the acrylic fibre. As a result a wide range of colours, in all depths, is obtainable on the wool and the wet fastness obtained compares favourably with that given on the acrylic fibre by basic dyes. Emulsifier OC is present as antiprecipitant for the anionic wool dye and cationic basic (Deorlene) dye.

The usual procedure is to prepare the dyebath with—

5–15% Glauber's salt	
2–3% Emulsifier OC	
3% Univadine W	(Univadine W calculated on weight of wool in the union, others on the total weight of the union)

2% Glacial acetic acid

Next the acid dye and the basic dye are added, both dissolved separately. The dyebath temperature is raised from 40–50°C to the boil in about 30 min and dyeing is continued for 1½ h at the boil.

For dyeing the wool component, Benzyl and Alizarine dyes are suitable, with the exception of—

Benzyl Fast Red 2RL
Benzyl Fast Blue R (C.I. Acid Blue 92)
Benzyl Fast Blue DB (C.I. Acid Blue 98)

1:2 Metal-complex dyes are not recommended as, apart from the possibility of the dye being retarded, precipitation may occur.

#### UNIONS OF ACRILAN AND WOOL (MILLING DYES)

Earlier recommendations have concentrated on the application of 1:1 (Neolan) and 1:2 (Cibalan) metal-complex dyes by conventional techniques. Milling dyes dye wool appreciably more deeply than Acrilan in unions. Reasonable solidity with many milling (Benzyl and Alizarine) dyes can be produced by pretreating the union with 2 g/l. sulphuric acid for 1 h at the boil (40:1 liquor ratio), squeezing without washing-off, and dyeing in presence of 3% Univadine W for 1 h at the boil. This method is of particular interest for scarlets, reds, and royal blues.

#### UNIONS OF ACRILAN AND NYLON

Interest has been shown in this type of union for the production of half-hose. It is preferable to dye the Acrilan first with modified basic dyes in presence of 3% urea and 2% of a non-ionic product, e.g. Emulsifier OC, for 1½ h at the boil, selecting basic dyes which show minimum staining of the nylon. If necessary, the stained nylon can be cleared by treatment with 1 g/l. potassium permanganate and 1 ml/l. glacial acetic acid for 20 min at 25°C, followed by clearing with 10 ml/l. sodium bisulphite (66°Tw.) for 20 min at 55–60°C. The nylon can then be cross-dyed with wool dyes (milling or metal-complex) in the normal manner. Chrome dyes are also of interest for deeper colours of the type required for half-hose.

#### UNIONS OF DYNEL AND ACRILAN

These two fibres have been used together in the manufacture of scatter rugs. Particularly if medium to full colours are desired, the easiest approach is to dye the two fibres separately. The following techniques merit consideration—

#### Disperse Dyes

These dyes vary in building-up properties and degree of solidity produced on Dynel and Acrilan. The following Cibacet dyes give reasonable results—

Cibacet Yellow GBA extra	(C.I. Disperse Yellow 3)
Cibacet Scarlet B	—
Cibacet Scarlet 2BW	(C.I. Disperse Red 1)
Cibacet Red E3B	(C.I. Disperse Red 15)
Cibacet Brilliant Pink 4BN	(C.I. Disperse Red 11)
Cibacet Sapphire Blue 4G	(C.I. Disperse Blue 16)
Cibacet Brilliant Blue BG	(C.I. Disperse Blue 3)
new	

Dyeing is carried out for 1½ h at the boil in presence of 1 g/l. of an anionic product, e.g. Emulsifier OC. Production of deep colours and the use of mixtures of dyes will present some problems.

#### Basic Dyes

When basic dyes are applied by the urea method (3% urea and 2% Emulsifier OC for 1 h at the boil), as is usual with Acrilan, the Acrilan dyes much more heavily than the Dynel. This effect can be prevented by adding a carrier, e.g. Invalon PR (0.5 ml/l.). Solidity is improved by increasing the

amount of Invalon PR used, but the Dynel tends to shrink and be impaired in quality.

### 1:2 Metal-complex Dyes

Reasonable solidity is obtainable with individual dyes of this type applied in presence of 8% sulphuric acid and 0.5 ml/l. of a carrier, e.g. Invalon PR for 1½ h at the boil. Suitable dyes include—

Cibalan Yellow GRL	(C.I. Acid Yellow 116)
Cibalan Brown VRL	—
Cibalan Grey 2GL	(C.I. Acid Black 62)

Larger amounts of Invalon PR are likely to have an adverse effect on the quality of Dynel. This process is limited to pale colours.

### CIBAPHASOL PROCESS

This is a continuous process developed for dyeing wool. Two aqueous phases are formed into an emulsion, sodium alginate being present as thickener and stabiliser for the emulsion. One phase (the coacervate) contains the dye and surface-active agent (Cibaphasol C), and the padded dye from this phase is deposited on and penetrates the material. Dye is fixed by steaming without intermediate drying or washing-off.

This process can be applied to wool-acrylic unions (e.g. with Courtele, Orlon, Acrilan 16) using wool dyes and modified basic dyes. Several factors must, however, be borne in mind, e.g. cross-staining of the wool by the basic dye and decomposition of some basic dyes.

### Dyeing of Zefran

Zefran differs fundamentally from other acrylic fibres in that it can be dyed with most of the classes of dye applicable to cellulosic fibres. 1:2 Metal-complex dyes from the main class of wool dyes of any value on Zefran. Disperse dyes are unsuitable because their dyeings on Zefran have very low fastness.

### DYES FOR CELLULOSIC FIBRES

To obtain an approximate evaluation of their suitability for dyeing Zefran, selected dyes from each class suitable for cellulosic fibres were applied to Zefran and to cotton, and the fastness to light and to washing of the dyeings were compared. The results are summarised in Table IV.

These examples show that dyeings of direct dyes have approximately the same fastness to washing on Zefran and on cotton, but lower light fastness on Zefran. Improved washing fastness is obtainable by appropriate selection, e.g. of dyes which give better wet fastness on cellulosic fibres by after-treatment with copper salts, formaldehyde, etc. A satisfactory standard of fastness to washing on Zefran can readily be obtained by using dyes of the sulphur, vat, and azoic classes and, by appropriate selection of dye, good light fastness is also obtainable. Although there will be individual differences, the standard of fastness obtained on cotton can generally also be obtained on Zefran.

### 1:2 METAL-COMPLEX DYES

When dyeing takes place at the boil, the effects obtained with dyes of this class are mainly limited to pale to medium depths. A reasonable standard of fastness to light and to washing is generally obtainable. Table V compares the fastness to light and to severe washing of Cibalan Grey BL (C.I. Acid Black 60) on Zefran, wool, and Acrilan. Zefran was dyed both in presence of ammonium sulphate (as for wool) and in presence of sulphuric acid (as for Acrilan).

As Zefran appears to withstand high-temperature dyeing satisfactorily, 1:2 metal-complex dyes have been applied by the method of BP 777,534, i.e. at 120°C with the addition of an electrolyte, e.g. common salt. Deep colours, including black, have been obtained by this method.

TABLE IV  
Fastness Properties on Zefran and on Cotton of Typical Dyes for Cellulosic Fibres

Dyeing	Dye Class	Colour Index No. C.I.	S.D.C. Washing Test No. 2 (3rd Report)						Light (daylight exposure)	
			Alteration	Staining of wool	Staining of cotton	Zefran	Cotton	Zefran	Cotton	Cotton
2.0% Chlorantine Fast Red K	Direct	Direct Red 81	1-2W	1-2W	4	3-4	1	1	2-3	4-5
2.0% Chlorantine Fast Blue GLL	Direct	Direct Blue 71	2-3W	3-4W	5	5	2-3	2-3	3	5-6
2.0% Chlorantine Fast Green BLL	Direct	Direct Green 26	3-4	3	5	5	3-4	3	2	5
6.0% Pyrogene Direct Blue BL	Sulphur	Sulphur Blue 11	4-5R	3-4W	5	5	4-5	3	3-4	4-5
2.0% Ciba Blue 2B powder	Indigoid Vat	Vat Blue 5	4-5	5	5	5	5	5	5	5
1.8% Cibacron Brown GR paste	Anthraquinonoid Vat	Vat Brown 3	4-5W	4-5R	5	5	5	5	5	5
2.0 g/l. Cibacaphthol RF	Azoic	Azoic Coupling Components 2 and 11	4	4	4	4	4-5	4	4	4-5

\* Substrate on which dyeing was prepared.

TABLE V  
Fastness Properties of Cibalan Grey BL on Zefran, Wool, and Acrilan

Substrate	Dyeing Method (1 h at the boil)	Fastness to Light (daylight)	Fastness to Severe Washing (S.D.C. Mechanical Washing Test A 3rd Report)		
			Alteration	Staining Wool	Cotton
Zefran	2% ammonium sulphate	4	4-5D	5	5
Zefran	8% sulphuric acid	4-5	4GD	5	5
Wool	2% ammonium sulphate	6-7	3W	4-5	5
Acrilan	8% sulphuric acid	5-6	3-4BR	5	5

I acknowledge my indebtedness to Ciba Clayton Ltd. for permission to publish this paper, and to several colleagues, in particular Mr. A. K. Taylor, Mr. D. G. Evans, and Mr. W. B. Harrison, for their valuable assistance.

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(MS. received 28th February 1961)

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- <sup>3</sup> Hadfield and Sokol, J.S.D.C., **74**, 629 (1958).
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### Discussion

#### SCOTTISH SECTION

Mr. J. B. SCALES: How can dyeings on acrylic fibres be stripped?

Mr. KRAMRISCH: This is a relatively difficult problem owing to the overall high standard of wet fastness obtained. Some success can be achieved with sodium chlorite or hypochlorite under acid conditions near the boil. The degree of stripping varies with the individual dye and depth of shade.

Mr. I. GAILEY: It would appear from the graph illustrating the influence of liquor ratio and salt on the uptake of Cibalan Black BGL at high temperature that a mathematical relationship can be deduced.

Mr. KRAMRISCH: This is possible but has not been examined.

Mr. G. D. BACON: Does acetic acid facilitate the application of 1:2 metal-complex dyes on acrylic fibres at high temperatures?

Mr. KRAMRISCH: Acidic conditions are useful with individual dyes.

Mr. J. G. B. MCCALLUM: Can disperse dyes be used for the acrylic component in unions of Courtelle and wool?

Mr. KRAMRISCH: Yes, but preferably only for light colours because of fastness problems. Furthermore, the possibility of cross-staining of the wool has to be taken into consideration in selecting a suitable dye.

Mr. GAILEY: How do Zefran and cotton compare for dyeing conditions and uptake?

Mr. KRAMRISCH: The application conditions are generally similar for the two fibres but we have not investigated dyeing rates.

Mr. BACON: How are unions of regular Acrilan and Acrilan 16 dyed?

Mr. KRAMRISCH: By using combinations of selected anionic (wool or reactive dyes) and modified basic dyes, using a one-bath process with Emulsifier OC as antiprecipitant.

Mr. J. WILSON: How are unions of Courtelle and cellulose acetate dyed?

Mr. KRAMRISCH: In pale depths selected disperse dyes may be satisfactory, but in medium and full depths it is necessary to combine modified basic and disperse dyes by a one-bath technique in presence of Emulsifier OC as antiprecipitant.

Mr. WILSON: How are Courtelle and Orlon bleached?

Mr. KRAMRISCH: Orlon—The manufacturers recommend in the first place the use of 3% formic acid (based on the weight of material) for 1½ h at the boil. A fluorescent brightening agent may be incorporated. Alternatively, the material may be treated with 1.5 g/l. sodium chlorite and 0.5 g/l. nitric acid (61%), raising the temperature slowly to 70°C and maintaining it at 70°C for 1 h.

Courtelle—The manufacturers do not recommend chlorite bleaching. A recommended method is treatment with 5% formic acid (on weight of material) and 1 g/l. Calgon T. The temperature is raised from 60°C to the boil in 30 min and maintained at the boil for a further 30 min. A fluorescent brightening agent may be incorporated. With both materials, slow cooling is advisable after bleaching to avoid crease marks.

#### WEST RIDING SECTION

Mr. C. L. BIRD: How does Zefran compare in physical properties with other acrylic fibres, particularly as regards steam pleating?

Mr. KRAMRISCH: There are both similarities and differences, but at this stage it is not possible to go into detail. Zefran is, however, suitable for pleating styles.

Mr. G. BRITTON: Is back-tanning required with blacks dyed with 1:2 metal-complex dyes?

Mr. KRAMRISCH: Although this treatment is often useful as an aftertreatment for dyeings on nylon, it is unnecessary on acrylic fibres.

Mr. H. BROADBENT: What are the main factors in controlling levelling with basic dyes on acrylic fibres?

Mr. KRAMRISCH: Temperature and pH control and the addition of Glauber's salt and proprietary retarding agents.

Mr. D. NEWSOME: Can acrylic fibres be mass-pigmented black?

Mr. KRAMRISCH: This is a commercial proposition.

Dr. H. B. HALLOWS: It has been suggested that basic and disperse dyes used together assist mutually in building up to fuller depths—has this been confirmed?

Mr. KRAMRISCH: Yes.

Mr. R. A. HAWKSWORTH: After stripping acrylic-fibre dyeings is it possible to redye the material to deep colours, e.g. black?

Mr. KRAMRISCH: Stripping of acrylic-fibre dyeings with chlorite or hypochlorite often appears to alter the dyeing properties of the material and in many cases it is extremely difficult to redye to deep colours.

Mr. A. K. TAYLOR: Stripping under acid conditions may result in a number of dyeing sites being occupied by hydrogen ions. In order to increase the number of available dyeing sites, the acidity of the fibre may be reduced by soap scouring.

Dr. C. B. STEVENS: A suggestion has been made elsewhere that disperse dyes will reproduce the majority of colours required on acrylic fibres.

Mr. KRAMRISCH: This is broadly true with certain reservations. As disperse dyes vary

considerably in their building-up properties on acrylic fibres, careful selection is essential. Furthermore, in certain cases the fastness achieved may not meet all trade requirements, particularly where the acrylic fibre is blended with wool and subsequently subjected to severe wet-processing treatments. Certain colours—mainly bright ones—cannot be reproduced with available disperse dyes.

Mr. GILBERT: Is it possible to dye acrylic fibres at low temperatures?

Mr. KRAMERISCH: At present the application of dyes to acrylic fibres at low temperatures does not appear to be a practical proposition. In particular the yield is much poorer than that achieved with dyeings prepared at a commercial boil.

## COMMUNICATION

# The Effect of Temperature and pH on the Adsorption of Basic Dyes by Wool

C. L. BIRD and G. P. STANCEY

Seven basic dyes have been applied to wool yarn in 0.5% depth at 40, 60 and 80°C, as well as over a range of pH values at 60°C. As far as wool is concerned, basic dyes appear to be rapid-dyeing dyes of low affinity, the exhaustion of which is very sensitive to change in pH in the region pH 4–6.

## INTRODUCTION

In the early days of the synthetic dyestuffs industry, basic dyes were of some importance on wool, as they enabled the dyer to produce brilliant shades which had hitherto been unobtainable. When acid dyes of similar hue became available most basic dyes were discarded, owing to their inferior fastness properties. At present, however, the behaviour of basic dyes towards wool is of some interest in connection with their use for dyeing unions of wool and certain acrylic fibres, e.g. Orlon (DuP) and Courtele (Courtaulds).

Although the effect of temperature and acidity on the exhaustion of basic dyes has been known qualitatively for many years, very little quantitative work has been published. Briggs and Bull<sup>1</sup> showed that the adsorption of Methylene Blue by wool at 100°C decreases below pH 6, becoming very small at pH 4.5. Similarly, Elöd<sup>2</sup> showed that the adsorption of Crystal Violet by wool at 90°C decreases steadily between pH 8 and pH 2. Bird and Firth<sup>3</sup> found decreasing adsorption of Chrysoidine by wool at 60°C between pH 6 and pH 2.5, but below pH 2.5 adsorption appeared to be unaffected by pH.

In view of this paucity of data it was decided to study the effect of temperature and acidity on the adsorption of basic dyes by wool, using dyes of different chemical constitution.

## EXPERIMENTAL

The wool used was in the form of 3/16s yarn spun from 64s dry-combed top. This was Soxhlet-extracted with diethyl ether and 64 O.P. ethyl alcohol, washed in distilled water, and conditioned at 65% r.h. and 22°C (moisture content 11.7%).

The dyes used were as follows. A pure sample of Chrysoidine hydrochloride (C.I. 11270) was supplied by Dr. J. M. Firth. Acridine Orange (C.I. 46005), in the form of the B.D.H. stain, was converted to the free base, and, in the dyeing experiments, the required amount of base dissolved in alcohol was added to an equivalent amount of oxalic acid in solution. Methylene Blue (C.I. 52015) was

obtained from Methylene Blue 2BN (ICI) by recrystallising several times from 64 O.P. alcohol and from water. Para Rosaniline (C.I. 42500) was obtained from Fuchsin (BDH) by recrystallising from water. Malachite Green (C.I. 42000) was supplied as the oxalate by I.C.I. Dyestuffs Division, and was recrystallised from water. Brilliant Green (C.I. 42040), supplied by British Drug Houses Ltd. (B.D.H.), was recrystallised from 10% aqueous acetone. Crystal Violet (C.I. 42555), also supplied by B.D.H., was recrystallised from water. In each case recrystallisation was continued until the optical density remained constant, and chromatography showed the presence of only one component. The dyes were stored in a room at 65% r.h. and 22°C.

Dyeings (0.5% on weight of yarn) were carried to equilibrium with 5-g hanks of humidified yarn in 500 ml of dye liquor in a Dyeometer (Ganz & Co., Zürich). In this instrument the dye liquor circulates through a glass cell forming part of a photoelectric absorptiometer, and the percentage transmission of the dye bath can be measured at any time without disturbing the dyeing. By reference to an appropriate calibration curve, the amount of dye adsorbed by the fibre can then be determined. Malachite Green was found to be strongly adsorbed on to the glass of the dye vessel, but was not adsorbed in the presence of wool. A little alcohol was used in preparing stock solutions of the dyes. Before dyeing, the yarn was wetted thoroughly, allowance being made for the water content of the wet yarn after squeezing.

The results obtained by dyeing at 40, 60, and 80°C are given in Table I, illustrated by Fig. 1. Activation energies of dyeing were obtained by multiplying the slope of the log  $t_d$  versus  $1/T$  graph by 2.3R. Fig. 2 and 3 show the effect on the equilibrium exhaustion at 60°C of varying pH, obtained by adding different amounts of sodium acetate and glacial acetic acid to the dye bath. The optical density of the dye solutions was not affected by change in pH over the range investigated.

TABLE I  
Times of Half-dyeing and Percentage Exhaustion at Different Temperatures

Dye	Temperature (°C)	$t_{1/2}$ (min)	Exhaustion (%)	Initial pH	Final pH	Activation Energy of Dyeing (kcal/mole)	$pK_a$
Chrysoidine	40	2.80	78	4.7	6.3	6.2	5.2† <sup>a</sup>
	60	1.66	71	4.7	6.5		
	80	0.96	65	4.7	7.3		
Acridine Orange	40	6.0	93	5.8	6.3	12.4	10.0† <sup>a</sup>
	60	1.6	90	5.8	6.4		
	80	0.65	85	5.8	6.4		
Methylene Blue	40	4.8	81	5.7	6.2	10.2	3.8† <sup>a</sup>
	60	2.0	69	5.7	6.1		
	80	0.76	50*	5.7	6.5		
Para Rosaniline	40	3.5	93	5.9	6.3	9.3	7.6 <sup>a</sup>
	60	1.6	89	5.9	6.5		
	80	0.7	82	5.9	6.9		
Malachite Green	40	7.6	94	4.3	6.5	9.3	6.9 <sup>a</sup>
	60	2.8	92	4.3	6.4		
	80	1.4	84	4.3	6.4		
Brilliant Green	40	15.5	90	4.6	6.1	11.1	7.9 <sup>a</sup>
	60	4.9	88	4.6	6.1		
	80	2.1	82	4.6	6.1		
Crystal Violet	40	9.5	97	5.5	6.2	12.5	9.4 <sup>a</sup>
	60	1.8	96	5.5	6.4		
	80	1.0	91	5.5	6.5		

\* Decomposition of dye occurred after 30 min.

† In 50% aqueous ethanol.

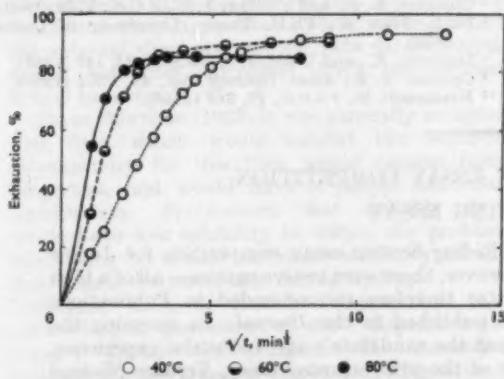


FIG. 1—Rate of dyeing of Acridine Orange

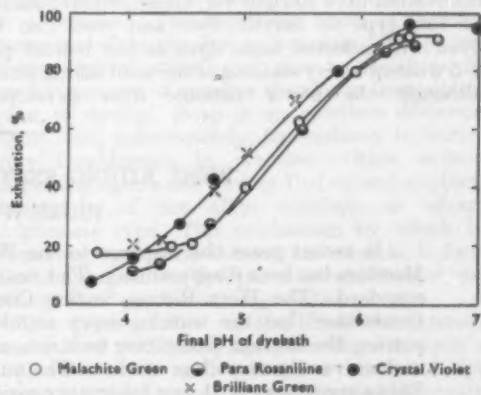


FIG. 2—Effect of pH on exhaustion at 60°C

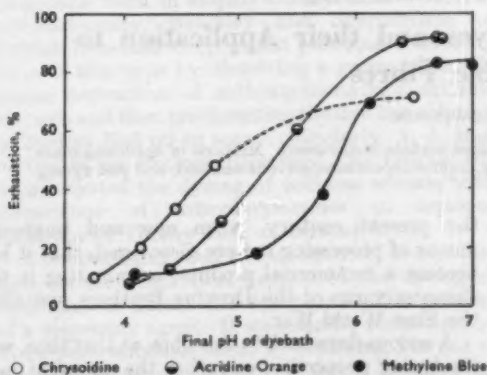


FIG. 2—Effect of pH on exhaustion at 60°C

It is well known that basic dyes aggregate in aqueous solution (see e.g. ref. 6). In the present experiments the percentage transmission of dye solutions was measured in the Dyeometer up to a concentration of 50 mg/l. On plotting optical density against concentration, a linear relationship, indicating no aggregation, was obtained with Chrysoidine. Para Rosaniline showed slight aggregation, and the remaining dyes considerable aggregation, at 40, 60, and 80°C.

#### DISCUSSION

Table I shows that at 80°C the times of half-dyeing ( $t_{1/2}$ ) are short, and the equilibrium exhaustions are rather low for this temperature. It may therefore be concluded that, in relation to wool, basic

dyes are rapid-dyeing dyes of rather low affinity which are suitable for application at relatively low temperatures. The low activation energies of dyeing support this view. It is noteworthy that corresponding acid dyes, e.g. sulphonated derivatives of triphenylmethane, often show poor exhaustion on wool from a dyebath containing the usual amount of sulphuric acid.

The sigmoid curves in Fig. 2 and 3 are mirror images of corresponding curves for acid dyes and can be explained on similar lines. The primary source of attraction in the wool is provided by the carboxyl groups. With fall in pH the wool acquires an increasing positive charge, which repels the positively charged dye ions. Affinity, however, will be due largely to non-polar van der Waals forces, which will cause the curve to be shifted to the left, the extent of the shift depending on the magnitude of these forces. The  $pK_a$  values given in Table I do not suggest that basicity is an important factor in connection with the affinity of basic dyes for wool.

The right-hand half of the curve for Chrysoidine in Fig. 2 is shown as a broken line, because in the pH region 5–6.5 it is almost certainly the free base which is being adsorbed.<sup>7</sup>

Compared with wool, acrylic fibres containing acid groups, e.g. Orlon and Courtelle<sup>8</sup>, have much higher affinity for basic dyes; and exhaustion of the dyebath appears to be less sensitive to pH—this is certainly the case<sup>9</sup> with the related fibre Acrilan 16. Consequently, unions of this type of acrylic fibre and wool can be dyed with selected basic dyes at the boil at pH 3–5 without heavy staining of the wool taking place, although the newer cationic dyes developed

specially for acrylic fibres are in general better in this respect than the classical basic dyes<sup>10</sup>.

It was hoped to extend this work by determining adsorption isotherms for basic dyes on wool, but the time available was sufficient for only one experiment to be carried out. With Crystal Violet, at pH 6.5 (disodium hydrogen phosphate buffer) and 60°C, an isotherm of the Langmuir type was obtained, which on extrapolation to infinite dyebath concentration gave a saturation value of 3.5 g dye per 100 g of humidified wool. The amount of dye on the fibre (loose wool) was obtained by difference from the percentage exhaustion of the Marney dyebath after 13 h.

\* \* \*

The authors thank the International Wool Secretariat for a scholarship which enabled one of us (G.P.S.) to take part in this work.

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING  
THE UNIVERSITY  
LEEDS 2

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## WEST RIDING SECTION ESSAY COMPETITION PRIZE-WINNING ESSAY

In recent years the response to the West Riding Section essay competition for Junior Members has been disappointing. This year, however, there were twelve entries—all of a high standard. The West Riding Section Committee therefore recommended to Publications Committee that the winning entry should be published in the *Journal*. In assessing the entries, the Section Committee took into account the candidate's age, industrial experience, and university or college studies. The author of the prize-winning essay, Terence Michael Baldwinson, who is 21, is a laboratory assistant who has been in the industry for four years; he is a first-year part-time student at Bradford Technical College.

### The Evolution of Disperse Dyes and their Application to Hydrophobic Fibres

T. M. BALDWINSON

The development of disperse dyes for dyeing cellulose acetate is discussed. Methods of application are then considered, attention being given to direct, carrier, high-temperature, solvent-assisted, and pad dyeing techniques.

#### Development of Disperse Dyes

"Necessity is the Mother of Invention". This is a well-known proverb, and its application to the textile industry is justifiable, as is particularly evident from the history of cellulose acetate fibre, and the frantic search for a method whereby it could be dyed. Cellulose acetate was known as early as 1870, but it is only since the beginning of

the present century, when new and improved means of processing it were discovered, that it has become a commercial product, culminating in the Celanese yarns of the Dreyfus Brothers just after the First World War.

A serious drawback to this fibre, at that time, was its dyeing properties, or rather the apparent lack of them, for it was impossible to dye cellulose

acetate by the then conventional processes with direct, acid, or basic dyes. In 1906 it was suggested<sup>1</sup> that this fibre could be dyed with acid or basic dyes in aqueous solutions of methylated spirit, acetone, or acetic acid, but this was expensive and gave very poor results, as regards both dyeing power and fastness properties. Subsequent proposals included the use of swelling pretreatments<sup>2</sup>, using mixtures of certain organic substances, the fabric being dyed afterwards in the usual manner with water-soluble dyes.

It was realised, however, that an ideal process would have to be "direct", that is, it should be possible to dye the fibre on conventional machinery with the minimum, if any, of dyeing assistants. At the beginning of the second decade, J. F. Briggs<sup>3</sup> prophesied that dye-makers would discover new dyes, specially developed for this fibre, whereby it would be possible to dye it by direct means.

Following this, the British Dyestuffs Corporation<sup>4</sup> developed the Ionamine series of dyes, which were *N*-sulphomethyl compounds derived by substitution at the primary and secondary amino groups of insoluble dyes. These dyes were the immediate precursors of the modern disperse dyes, and represented a definite advance over all previous types, but they suffered from the severe limitations brought about by the necessity to make them soluble in water, which was essential if dyeing was to take place. Difficulties also occurred when mixtures of these dyes were used, for the process depended upon the fact that, under dyeing conditions, these compounds hydrolysed to produce the original dye, and as the rate of hydrolysis varied with the individual dye<sup>5</sup>, trouble was bound to occur.

About this time (1923) it was generally accepted that dyes which would exhibit the required substantivity for this fibre would possess basic properties, and would have a simple molecular constitution. It seemed that suitable dyes would have low solubility in water, the problem then being to obtain sufficient solubility of the dye, which was necessary during dyeing.

Holland Ellis, of British Celanese Ltd, the acknowledged discoverer of the "disperse technique", solved this problem. He succeeded in producing fine aqueous suspensions (dispersions) of a number of water-insoluble azo dyes by dispersing them in sulphuric acid<sup>6</sup>. Almost simultaneously, Baddiley and Shepherdson of British Dyestuffs Corporation produced a series of dyes of this type by dissolving a group of simple amino derivatives of anthraquinone in a suitable solvent, and then precipitating the dye in a solution of Turkey Red oil or soap<sup>7</sup>. Similarly, A. J. Hall of the Silver Springs Bleaching and Dyeing Co. demonstrated the dyeing of cellulose acetate with dispersions of nitrodiarylamines in aqueous solutions of Turkey Red oil or soap<sup>8</sup>.

It is interesting, and of consequence, to consider here certain theoretical aspects of the dyeing of hydrophobic fibres, and to examine the purpose of a dispersing agent. If we consider viscose rayon to be pure cellulose, then, by replacing five out of the six hydroxyl groups by acetyl groups, we obtain secondary cellulose acetate, the acetyl

groups amounting to about 40% by weight of the fibre. As a result of this, the fibre has a much reduced affinity for water, and it swells to a much smaller extent than cellulose in aqueous solutions—hence the lack of affinity of this fibre for the water-soluble dyes. Also, the average pore size of cellulose acetate is about 10–20 Å, whereas viscose rayon has a pore size of 20–30 Å<sup>9</sup>. Therefore, in order to penetrate the intermicellar spaces, a suitable dye should possess a simple molecular constitution, although, as a general rule, the smaller the dye molecule, the lower is its fastness to light and to washing.

It is possible to dye cellulose acetate with disperse dyes without the use of a dispersing agent, but such a method results in speckly dyeings of poor fastness, because small particles of dye adhere to the surface of the fabric<sup>10</sup>. The purpose of the dispersing agent, then, is to promote the levelling properties of the dye, and to prevent dye particles from adhering to the fabric. Having considered the purpose of the dispersing agent during the dyeing process, we must now examine its use during the manufacture of the dye. The final stage in the manufacture of disperse dyes involves the reduction of particle size to less than 4 μ, by means of a milling process in which the dye slurry is rotated in a ball mill containing flint stones or pebbles together with the dispersing agent. Milling effectively reduces the particle size, but without the agent the dye would quickly aggregate during the subsequent process of drying to a powder. The dispersing agent is therefore incorporated in the dye to maintain the dye in a finely divided state, and also to facilitate the reverse process of aqueous dispersion immediately prior to dyeing. Soap is an excellent dispersing agent, but, unfortunately, its tendency to froth is very troublesome in practice. Other suitable dispersing agents are Turkey Red oil and synthetic detergents of the alkyl sulphate or alkaryl sulphonate type. The mechanism by which the dispersing agent acts is electrical: that is, it forms like charges on the dye particles which repel each other, thus preventing aggregation.

Two types of dye resulted from the intensive research of the 1920s; first, the disperse dyes, as outlined above, and, second, a series of dyes known as the Duosols<sup>11</sup>, which were sulphonated or sulphated dyes soluble in water or aqueous solutions of organic compounds. Both types had similar fastness properties, but the Duosols were sensitive to slight variations in the physical characteristics of the fibres, and so gave uneven dyeings owing to selective absorption<sup>11</sup>. As a result of this, it was not long before the Duosols were dropped from the range of dyes for cellulose acetate.

Before passing on to describe the methods of application of this particular class of dye, it will be useful first to examine the behaviour of mixtures—an important matter where jig dyeing is concerned. Individual disperse dyes have different rates of dyeing, and when mixtures of dyes are used in jig dyeing (say a blue and a yellow to produce a green) it is essential that the components should have the same rates of dyeing at and below

the dyeing temperature, otherwise "listing" will occur. For example, suppose it is desired to produce a green on Tricel fabric. If the blue component dyes considerably slower than the yellow, the green hue will not be maintained, owing to listing of the blue. If, however, a slower-dyeing yellow is used (either in conjunction with the quicker-dyeing yellow or alone) it will be possible to maintain the green hue throughout dyeing—this constitutes a satisfactory mixture. Tricel is almost invariably dyed near the boil (95°C), but it must be remembered that the temperature of the jig rollers, where much of the dyeing takes place, will be much lower (say 85–80°C). Hence any mixture used must consist of components which dye at equal rates at 95°C and at 80°C.

Having thus considered the history and a certain amount of the theory of disperse dyes, it will now be convenient to discuss the various methods of using them.

### Application of Disperse Dyes

Usually, when considering a new dye, the dyer's first thought is: "How easy is this dye to apply?" This, indeed, is of primary importance, for the ease, or otherwise, of application governs the always-overriding factor of cost; dyes which are difficult to apply result in an increase in labour and materials, and possibly expensive machinery. Secondly, the dyer gives consideration to the fastness properties of the dyes, for these govern to a large extent the usefulness and life-span of the dyed fabric. In turn, these two factors when combined (cheapness and durability) govern the selling-ability of the fabric.

Do the disperse dyes, then, come up to the dyer's standard? In general they do, for, apart from one or two cases where it is advantageous to use assistants or temperatures above 100°C, the disperse dyes can be dyed from the usual winches and jigs found in the normal dyehouse, and are as easy, even if somewhat slower, to apply as direct dyes to cellulosic fibres. The fastness properties of individual dyes do, of course, vary considerably, and it is particularly unfortunate that the faster dyes, which have a greater molecular complexity, have inferior dyeing power. Even so, it is usually possible to effect a reasonable compromise between dyeing power and fastness properties, for today the dyer has at hand a large and ever-increasing selection of disperse dyes of excellent fastness and good dyeing power.

The disperse dyes were originally made for dyeing secondary cellulose acetate, and the process proved to be highly beneficial, for no change in dyehouse routine was required, the process being very simple and direct. Most of the cellulose acetate dyed today is winch- or jig-dyed in the form of pieces, for the disperse dyes do not provide the necessary fastness to wet-processing which is demanded of yarn-dyed goods, although yarn which is intended for fabrics finished in the loom state may be dyed before weaving.

With the advent of synthetic-polymer fibres (polyamides, polyesters, and acrylics), all of which

proved admirable substrates for disperse dyes, an impetus was given to the industry to produce even more dyes, specially designed for specific fibres.

In all, there are five important methods of dyeing hydrophobic fibres. Each of these methods, with their advantages and disadvantages, will now be considered individually.

### DIRECT DYEING

In this method, the dyebath contains only the dye and the requisite amount of dispersing agent (soap or other suitable substance) in water. It is eminently suitable for secondary cellulose acetate and nylon, and for light to medium depths on cellulose triacetate (e.g. Tricel) and acrylic fibres (e.g. Orlon 42, Courtele, Acrilan).

Secondary cellulose acetate is dyed for about 15 min at 60°C, after which the temperature is raised to 80°C, and dyeing continued for a further 45 min. On no account must the temperature be allowed to exceed 85°C, otherwise serious delustering of the fabric will occur. In the case of fabrics dyed on the jig, particularly when mixtures of dyes are used, it is advisable to use a covered jig so as to reduce the amount of cooling on the jig rollers, and so encourage the individual components to dye at equal rates, otherwise listing of the slower-dyeing components will occur.

Perhaps one of the greatest faults of cellulose acetate is its notorious tendency to exhibit "burnt-gas fume" fading, which was particularly evident in the earlier days of the disperse dyes. Anthraquinone dyes which contain amino groups are the trouble-causers; they include most of the blues, some violets, and some reds. This phenomenon seems to be caused by the nitrous oxide present in burnt-gas fumes, which reacts with the amino groups of the dye<sup>5</sup> in much the same way as nitrous acid reacts with the well-known diazotisable dyes. However, there now exists a number of dyes, including blues, which are resistant to nitrous oxide, a typical example being Serisol Fast Blue BGLW. Even so, the range of fast blues is limited, and gas-fume-fast dyes are, in general, expensive. It is often necessary, therefore, to make use of inhibitors, which are, in effect, colourless disperse dyes which adsorb the burnt-gas fumes preferentially, hence allowing use to be made of the cheaper, non-fast dyes. They are applied in just the same way as the disperse dyes, either together with the dye or, preferably, after the dye. Typical examples of commercial inhibitors are Antiacid 3546 (Courtaulds), Inhibitor GFN (BASF), and Protac 30 (YDC). They do not, however, completely solve the gas-fume-fading problem, for they do eventually wash out of the fabric.

It is unfortunate that, as the fastness properties of the dye are improved, there is a corresponding decrease in dyeing power<sup>5</sup>; this is true of both fastness to light and fastness to washing. Whilst many disperse dyes exhibit good fastness properties on cellulose acetate, the majority do not possess sufficient fastness to be able to withstand much wet-processing, and hence, the majority of acetate fabrics, which are mainly used for clothes

linings, dress and lingerie fabrics, are dyed in the piece. One of the most attractive uses of cellulose acetate is in furnishing fabrics in which it is mixed with either viscose rayon or cotton. By dyeing the acetate with disperse dyes and the cotton or viscose rayon with direct dyes, both in the same bath, many interesting and attractive cross-dyed effects can be produced.

As previously stated, the direct method is suitable for the dyeing of nylon, although it is safe, and customary, to use a higher temperature (85–90°C for 1 h) than with acetate. Ladies' nylon stockings, however, are generally dyed for 2 h at 60°C, it being essential that the rate of uptake of the dye be low, in order to ensure that the higher-denier yarns, which form the tops of such articles, will dye at approximately the same rate as the lower-denier yarns forming the bulk of the article.

Disperse-dyed nylon is not, in general, sensitive to burnt-gas fumes, but the wash- and light-fastness of the dyeings are lower than on secondary acetate, and much inferior to those of dyeings produced on nylon by acid, metal-complex, or chrome dyes, though these three classes of dye are difficult to apply, and are also sensitive to slight variations in the physical structure of the yarn<sup>12</sup>. Nor is it necessary, in most cases, to choose dyes of high fastness to sublimation, for the majority of nylon piece goods are heat-set prior to dyeing. Some nylon is also dyed by the high-temperature method (120°C), but it is doubtful whether the shorter dyeing time (30 min) is sufficient to compensate for the high cost of machinery and the extra steam required.

Where only pale to medium depths are required, this method is also suitable for Tricel, a higher temperature being employed (95°C) for about 90 min.

Polyacrylonitrile fibres (e.g. Orlon 42, Courtele, Acrilan) provide yet another substrate for disperse dyes; these, too, are dyed by the direct method, usually at or near the boil for 2 h. Although it is impossible with the disperse dyes to produce heavy depths (for which the basic dyes are usually used), the disperse dyes will produce pale to medium depths of adequate fastness, and they also possess better levelling properties than do the basic dyes, to say nothing of ease of application. The use of carriers is not an economic proposition, nor are higher temperatures, for the affinity of the polyacrylonitrile fibres for the disperse dyes is low, and carriers (and elevated temperatures) only increase the rate of absorption, and not the amount. Great care must also be taken over temperature control when dyeing acrylic fibres, owing to their thermoplastic nature, and it is necessary to allow them to cool slowly in the dye liquor to about 60–70°C before removing them from the bath.

It will be seen, then, that the direct method has many uses, and readily adapts itself to the dyer's requirements, owing to its simplicity. This is the method used for colouring many plastics, including nylon toothbrush handles and polyester buttons.

#### CARRIER DYEING

Some fibres, notably cellulose triacetate (e.g. Tricel) and polyesters (e.g. Terylene), are capable of absorbing large quantities of disperse dyes, but they do so at very low rates, and, because of this, it would not be a practical proposition to dye them by the normal direct method, particularly where heavy depths are required.

Dyes with small molecules dye more quickly, but they cannot be used where high fastness is required, for just as they are easy to apply, they are equally easy to remove when the fabric is subjected to heat and wet treatments<sup>13</sup>. Fabrics made from Tricel and Terylene, which are well known for their crease-resisting properties, are almost invariably subjected to such treatments. And so, once again, the research laboratories were feverish with action aimed at finding a suitable means of increasing the rate of dyeing with the larger-molecule dyes.

Many organic compounds will do this, typical examples being diethyl phthalate, and *o*- and *p*-phenylphenol. Such compounds are added to the dye bath, and are termed carriers, two commercially available carriers being Dicene 60 (YDC) and Latyl Carrier X (DuP). The amount of carrier used depends on the depth required, but is normally small (e.g. 2–8 ml/l. of dye liquor), and dyeing is usually complete after 1½–2 h at or near the boil. The success of a carrier depends largely on its ability to associate with the polymer chains of the fibre (not with the dye)<sup>14</sup>. Carriers also act as softening agents, opening up the fibre structure and making it more accessible to the dye, which is to a certain extent soluble in the carrier, and so the rate of absorption is increased.

Trouble may, and sometimes does, occur with this method of dyeing, especially with volatile carriers which tend to condense in the atmosphere and fall back into the liquor, causing a "spot" on the fabric, such spots being impossible or extremely difficult to remove. And, of course, volatile carriers, which often have an unpleasant odour, can be injurious to the health of personnel employed, to say nothing of the additional expenditure involved in buying carriers.

This method is of special importance for dyeing Tricel, and so special consideration will be given to this fibre. In common with the polyester fibres, Tricel has to withstand thermal finishing operations, and these usually have to be carried out after dyeing, because heat-setting reduces the affinity of the fibre for disperse dyes. Terylene, on the other hand, is only slightly affected, and in some cases the affinity is actually increased. This phenomenon is put to use in the dyeing of Terylene-Tricel blends, where normally the Tricel would dye preferentially to the Terylene. It follows that it is essential to have available dyes which are unaffected by heat treatments, i.e. that are fast to sublimation.

A considerable quantity of Tricel is also subjected to the process of surface-saponification (with caustic soda), in which a surface layer of cellulose triacetate reverts to cellulose. This process, too, reduces the affinity of the fibre for the dye. It is

therefore necessary in the case of deep colours, such as navy blues and blacks, to dye prior to the saponification process, and this renders it essential to have at hand dyes which are stable to hot solutions of caustic soda (of approx. concentration 3–4 g/l.). The advantages to be gained from surface-saponification are numerous and far outweigh the disadvantage of decreased affinity for dyes; for example, whereas a particular dye on Tricel may be sensitive to burnt-gas fumes, the same dye on saponified Tricel will be largely resistant to fading under these conditions<sup>15</sup>, and it may be possible to dispense entirely with an inhibitor, or, at any rate, to use a much smaller quantity.

The precautions used when dyeing secondary cellulose acetate must also be observed when dyeing Tricel on the jig with mixtures of dyes. According to Fourness<sup>5</sup>, the light fastness of dyeings on Tricel is slightly less than on secondary acetate, but the wash fastness is better.

Terylene, too, may be dyed by the carrier method, although it is not necessary to pay attention to such factors as gas-fume fading, effect of saponification, and the preferential absorption of the various components from a mixture of dyes.

#### HIGH-TEMPERATURE DYEING

It was stated above that for dyeing Terylene, it was essential to find some means of increasing the rate of absorption of the larger-molecule dyes, and that carriers successfully did this. However, many carriers have an adverse effect on the light fastness of these dyes on this fibre<sup>12</sup>. *o*-Phenylphenol, for instance, slightly lowers the fastness, whilst *p*-phenylphenol is even more severe in its action<sup>16</sup>. There is also, as was pointed out previously, the added disadvantage of unpleasant smell, volatility, and expense of carriers. Hence, there was a need for another method of dyeing Terylene in which carriers could be dispensed with, and this was met by developing the method of dyeing at temperatures above 100°C under pressure. For Terylene, the optimum temperature is 130°C<sup>18</sup>, which necessitates a pressure of 25 lb/in<sup>2</sup>. This is by far the best method yet devised for dyeing polyester fibres; it cuts the dyeing time from 2 h to 30 min and sometimes results in better fastness<sup>20</sup>, whilst more level results, and hues of greater strength and brightness, can be obtained. The method is sometimes used for dyeing nylon (120°C) and Tricel (125°C).

The theory of high-temperature dyeing is very much the same as that of carrier dyeing: heat increases molecular activity and opens the structure of the fibre, thus facilitating diffusion of the dye molecules into the fibre<sup>14</sup>.

Unfortunately, the pressurised jigs and machines of the liquor-circulation type required for this work are expensive and are not economical unless a considerable volume of work is to be undertaken. Despite this, the method seems to be gaining in popularity, which is proof enough of its value.

Polyester fibres possess considerable strength, and hence serve a useful purpose in suits and other

clothing in which they are often blended with wool. In this capacity, they are likely to be subjected to the rather drastic treatment of thermal finishing or heat-pleating operations, which necessitate the selection of dyes with a high degree of fastness to sublimation. Such union fabrics are dyed by the carrier method (high temperatures have a very drastic effect on the wool), and it is essential to choose dyes which do not stain wool, and also to choose a carrier which helps in this direction (e.g. Seripolan (YDC)), otherwise a two-bath method will have to be used, the wool being cleared in a reduction bath after dyeing the Terylene, before being dyed itself.

#### SOLVENT-ASSISTED DYEING

If a compound such as benzyl or butyl alcohol is added to the dyebath, it is found that the time required to produce a particular depth is considerably less than when no alcohol is added. This is because the dye is soluble in such compounds and because hydrophobic fibres swell in the presence of these solvents, thus allowing the dye to penetrate more quickly into the intermicellar spaces. Gokhale, Peters, and Stevens demonstrated this effect with Duranul Red X3B.300 on Tricel, and found that with 77 g/l. of *n*-butanol in the bath, 80% exhaustion was obtained in 15 min at 80°C, and in 30 min at 40°C<sup>18</sup>. They also showed that equilibrium was reached at 40°C after 2 h, whereas 240 h at this temperature was not sufficient in absence of alcohol. As well as a considerable reduction in the dyeing time, much stronger and brighter dyeings are obtainable by this method.

Unfortunately, this too is an expensive process, it being necessary to install apparatus in order to recover the alcohol. As a consequence this process has not found general acceptance amongst dyers, although it is reputed to be used more often in the U.S.A. than in the British Isles. It is a method which is suitable for all the hydrophobic fibres except secondary acetate (which can easily dissolve in the solvents used), but commercially it is reserved for those fibres which under normal conditions have a long dyeing period, e.g. Terylene, Tricel, and the acrylic fibres.

#### PAD-DYEING

This is another process in which the dyeing time is considerably reduced. Briefly, the process consists in soaking the fabric in a viscous dispersion of the dye (e.g. in a solution of sodium alginate) and then padding the dye on to the fibre by passing it through a mangle, afterwards washing off excess of dye and alginate, drying, and fixing the dye in dry heat (say  $\frac{1}{2}$ –2 min at 200°C). A serious drawback to this process is the difficulty, if not the impossibility, of carrying out accurate matchings. Furthermore, the mangle used must be mechanically perfect, otherwise unlevel dyeings will ensue; and, of course, the dyes used must be capable of withstanding the high temperatures used in the dry heat-setting process. This method is suitable for Tricel, Terylene, and nylon.

#### Conclusion

It is now almost half a century since the first disperse dyes were born out of the necessity which

arose for them, and there is still, and probably always will be, considerable scope for research into both the disperse dyes themselves and the methods of applying them. It is still necessary to continue working for better fastness properties, particularly in the case of the wash-fastness of dyeings on nylon, and as new fibres come along, it is usually found necessary to produce even more dyes which will exhibit the required fastness properties on that particular fibre.

The disperse dyes are now established as an important and integral part of the textile industry, and are used in increasing quantities every year. To quote Fourness<sup>5</sup>, without disperse dyes and the disperse technique, "many man-made prodigies would have been stillborn, or at best remained Peter Pans".

[EDITOR'S NOTE—The essay was illustrated by several dyeings which it was not feasible to reproduce in the *Journal*]

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## CORRESPONDENCE

*The Editor does not hold himself responsible for opinions expressed by correspondents*

### Observations on the Identification of Acrilan, Acrilan 16, and Courtele

The acid dye Orange II may be used to distinguish between Acrilan and Courtele because of the greater affinity of Acrilan for acid dyes. However, from enquiries we have received, it would appear that there is still difficulty in identifying these two fibres, and now, with the introduction of Acrilan 16, it is necessary to differentiate between three similar materials. A summary of the tests carried out in this laboratory to solve the problem is now given.

Before applying these tests it is, of course, necessary to ascertain by a standard procedure that the fibres under test are in fact acrylic<sup>1</sup>.

#### MICROSCOPICAL TESTS

Acrilan 16 may be distinguished from the other types by its definite bean-shaped cross-section. There is, however, a difference between normal Acrilan and Courtele which appears to be the basis of the method used by several laboratories to distinguish between them. This difference is in number and size of delustrant particles within the fibres, those in Courtele being more numerous and

more finely dispersed than those in Acrilan (see Fig. 1).

#### STAINING TESTS

A table is given below, listing the effect of Orange II, Rhodamine B, and Shirlastains A and C on each of the fibres under specified conditions.

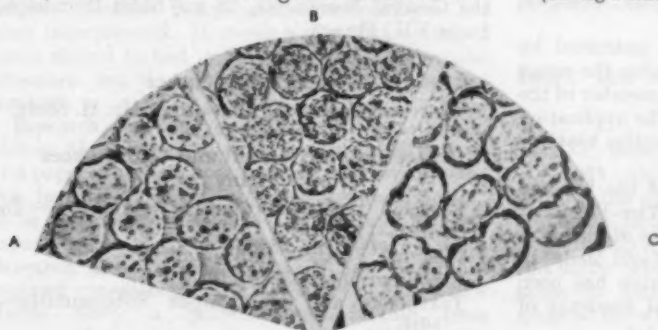
Treatment <sup>a</sup>	Acrilan	Courtele	Acrilan 16
3% Orange II <sup>a</sup>	Intense orange	Very faint flesh pink	Flesh pink
2% Acetic acid			
10% Glauber's salt			
3% Rhodamine B <sup>b</sup>	Deep pink	Magenta	Magenta
1% Acetic acid			
Shirlastain A <sup>c</sup> (cold)	Pale dusty purple	Virtually unstained	Very pale dusty mauve
Shirlastain A <sup>c</sup> (boiling 5 min)	Brown olive (Lovat)	Virtually unstained	Fawn (light yellow-brown)
Shirlastain C <sup>c</sup> (cold)	Very pale lilac	Pale lilac	Lilac
Shirlastain C <sup>c</sup> (boiling 5 min)	Dark green	Light green	Dark green

<sup>a</sup> Liquor ratio 100:1; bring to boil in 30 min, boil for 30 min, wash well.

<sup>b</sup> Liquor ratio 100:1; raise to 85°C in 30 min, maintain at 85°C for 45 min, wash well.

<sup>c</sup> The Shirlastain tests are included because, contrary to general belief, it is possible to differentiate between the three fibres without resort to microscopy or any other test. It is advisable to test authentic samples for comparative purposes and to use all four Shirlastain tests if complete confidence is to be had in the conclusions. Except for the time of boiling, the Shirlastain is used according to the recommendations of I.C.I. Ltd.<sup>2</sup>

<sup>d</sup> These tests are applicable only to undyed material.



A Acrilan, 3 den. B Courtele, 3 den. C Acrilan 16, 3 den.  
FIG. 1 (×400)

## SOLVENT TESTS

The following procedure is an extension of the tests recommended by the Textile Institute<sup>3</sup>.

Treat the material with dimethylformamide—

- (a) At 20°C Courtelle dissolves in 15 min.  
Acrilan 16 is not completely dissolved after 35 min.
- (b) At 55°C Acrilan dissolves in 8 min.  
Acrilan 16 dissolves in less than 30 s.

It should be stressed that tests have not been carried out on an extended range of dyed materials

and it is possible that certain types of dye may affect the solubility of the samples.

J. T. BALL

DEPARTMENT OF TEXTILE INDUSTRIES  
INSTITUTE OF TECHNOLOGY  
BRADFORD 7

23th March 1961

<sup>1</sup> *Identification of Textile Materials* (Manchester: The Textile Institute) 4th Edn. 1958, pp. 58–59.

<sup>2</sup> *Shirlastains A & C for the Identification of Textile Fibres*, I.C.I. Technical Circular, 1954.

<sup>3</sup> Reference 1, p. 43.

## Notes

### Bristol Symposium, 1961

The provisional programme for this symposium, which is to be held in Bristol on 20th–22nd September 1961, includes papers on the coloration of textiles (acrylic fibres, nylon, wool, package dyeing of yarn), leather, and plastics, diffusion of dyes in polymer films, colour measurement and matching (in the paper, paint, and textile industries), and stability of reactive dyes. Further details will be available shortly.

### Canadian Association of Textile Colourists and Chemists

On 21st April 1961, Mr. J. G. Soroka was re-elected President of the C.A.T.C.C., and Mr. S. H. Chambers Honorary President. The Vice-Presidents for the coming session are Mr. J. H. Lowe and Mr. C. R. Teichgraber. Mr. T. R. Owen and Mr. D. Heggie were re-elected Secretary and Treasurer, respectively. Association Councillors are—Messrs. P. W. Benn, F. Martin, K. Hogan, A. G. Plumb, A. Zinnator, and F. J. Deegan.

### 1961 Mattiello Memorial Lecture

The Federation of Societies for Paint Technology announces that Dr. Louis Arnold Jordan, Founder Director of the Paint Research Station, Teddington, and of the Research Association of British Paint, Colour and Varnish Manufacturers, will present the 1961 Joseph J. Mattiello Memorial Lecture at the 39th Annual Meeting of the Federation to be held in Washington, D.C., on 2nd–4th November 1961.

The Mattiello Memorial Lecture, instituted by the Federation in 1949, commemorates the name of Dr. Joseph J. Mattiello, who, as a member of the Federation, did so much to expand the application of science in the decorative and protective coatings field.

Dr. Jordan has been President of the Oil and Colour Chemists' Association, and Vice-President and Chairman of Council of the Society of Chemical Industry; he received the Society's Gold Medal in 1953. Since January 1958, Dr. Jordan has been Professor of Chemistry at the Royal Academy of Art, London.

The provisional title of the lecture is "Science and Society—Master or Servant".

### National Lending Library for Science and Technology

In the April issue of *LLU News*, the news-sheet of the DSIR Lending Library Unit, it is reported that, with the establishment of the National Lending Library for Science and Technology at Boston Spa, Yorkshire, the Lending Library Unit is being wound up. The Reading Room at Chester Terrace, London N.W.1 is now closed; it is likely that the Reading Room at Boston Spa will be open at the beginning of September. From this time visitors will be welcome at Boston Spa; the hope is expressed that organisers of conferences of scientists, technologists, and librarians in neighbouring towns will consider organising visits.

### Second Effluent and Water Treatment Exhibition and Convention

This exhibition will be held at The Seymour Hall, London W.1 on 31st October–3rd November 1961. Papers on effluent and water treatment will be presented. Information is available from Dale Reynolds Publicity, 2 Broad Street Place, London E.C.2.

### Conference Internationale des Arts Chimiques

The VIth Salon Internationale de la Chimie and the Conference Internationale des Arts Chimiques will be held from 25th April to 4th May 1962 at the Centre National des Industries et des Techniques (C.N.I.T.), Rond-Point de la Défense at Puteaux-Paris. Details of these events are obtainable from the General Secretariat, 28 rue Saint-Dominique, Paris VII, France.

### Death of Member

We regret to report the death of Mr. H. Stott.

### Meetings of Council and Committees May

Fastness Tests Co-ordinating Committee—2nd Council—3rd  
Washing Fastness Subcommittee—3rd  
Examinations Board—16th  
Textbooks and Monographs Subcommittee—16th  
Publications—16th  
Colour Index Editorial Board—24th

## OBITUARY NOTICE

### Lance Barford

The sudden death on February 3rd of Lance Barford, after a lecture given to the London Section of the Society, was a great shock to his large number of friends and acquaintances, although he had been in indifferent health for two or three years.

Lance Barford was educated at Bedford School. In 1920 he became a student in the Colour Chemistry and Dyeing Department at Leeds University, leaving in 1924 to join the family firm of Barford Bros. Ltd in Luton. With his brother, R. M. Barford, he controlled the dyeing and finishing activities of the firm up to the time of his

death. The firm, originally straw dyers and bleachers, later branched out into the processing of ribbons and felts.

Barford joined the 5th Battalion Beds. and Herts. Regiment, T.A., in 1926 and served throughout World War II both with his Battalion and with the Corps of Military Police Traffic Control, holding the rank of Major. He saw service in France, Belgium, Holland, and Germany, and was awarded the Territorial Decoration.

His loss will be felt severely, not only by his personal friends, but also by the dyeing and finishing industry in Luton. F. L. FACE

## New Books and Publications

### Review of Textile Progress

#### Volume XI—1959

Manchester, Bradford, and London: Textile Institute, Society of Dyers and Colourists, and Butterworths. Pp. x + 448. Price, 60s. Od. (45s Od. to members of the Institute or the Society).

To review a review is always difficult, and this difficulty is enhanced when the Society has contributed to the volume in question. The reviewer must, therefore, condense the uncondensable, and, if need be, decry a Society publication in the Society's journal. Fortunately, the *Review of Textile Progress* in this, the eleventh volume, reaches the same clear, concise excellence as its predecessors. Every account in the volume is a useful compendium of references to most of the work done in the textile field throughout the year.

The major differences from previous volumes are in the regrouping of some sections. Thus "Chemistry and Physics of Fibrous Materials" and "Fibre Production" are now combined under the heading of "Production and Properties of Fibres". This large section is then subdivided into three subsections dealing respectively with cellulosic, protein, and synthetic-polymer fibres. "Colouring and Finishing" are now grouped under "Chemical and Other Processes", whilst an entirely new section "Chemical Compounds other than Dyes" has been incorporated. It seems a pity that dyeing of fibres should be lost in the vague title of chemical processes, but though lost in name the substance remains.

Research on the production of acetate and viscose fibres has resulted in a number of patents and papers this year, particularly interesting being the investigations of catalysts for acetylation. It is noticeable that work on cuprammonium rayons seems to have almost completely ceased. Research into the preparation of new fibres has declined considerably, the advent of only one new elastic fibre, a polyurethane, having been announced this year. Even the prolific acrylics have settled down and there are no new fibres to puzzle and harass the processors. More emphasis

has been placed on modification of known materials and improved production techniques, interfacial condensation polymerisation receiving considerable attention. Possibly we can hope that the continual introduction of new fibres has now terminated and that manufacturers are concentrating on those of proved value. It is worth noting that Russia and Japan continue to develop their polyamide and polyvinyl alcohol fibres, respectively.

In fibre physics research appears to be concentrated on the fine structure of fibres, particularly crystallisation phenomena and the morphology of single crystals.

No major development is reported in yarn and fabric production, nor in raw material purification, the work being mainly concerned with improvements in established methods.

The chapter on colouring matters this year deals solely with reactive dyes, and the mass of new data on this subject is evidence of the tremendous interest in this relatively new field.

The new chapter on "Chemical Compounds other than Dyes" contains much information on research into antistatic, water-repellent, and flame-proofing agents, sizes, bactericides, and fungicides. It is a very welcome addition to the volume.

Research work in widely different fields of textile technology is adequately covered and workers will find this book a valuable collection of references to the year's patents and papers.

R. S. ASQUITH

### Index des Hules Sulfonées et Détergents Modernes Volume III

By J.-P. Sisley. Pp. 560. Paris: Editions Teintex. 1961. Price, 80 NF.

Since the indexing of dyes has, for the present, reached a satisfactory state, attention appears to be currently turning towards the classification of speciality chemicals; a number of very laudable attempts have recently appeared. Sisley has adopted the wise course of attempting to cover only a portion of the field and to cover that thoroughly. As with dyes, classification of speciality chemicals by constitution or by application is possible.

Most attempts at codification of the very wide ranges of products which fall under this rather loose heading are based on application, but when only surface-active agents are under consideration classification by constitution is more apt. Even in this narrower field the difficulties encountered are at least as great as with dyes, and an index of this nature compiled by an author particularly well placed by profession is probably as far as it is possible to go.

According to the table of contents, the book is divided into seven chapters, and these are given as—Introduction; Abbreviations; Development and production; Synthetic surfactants; Classification; Index of commercial brands; Alphabetical descriptive index. Some confusion seems to have occurred, since Chapter 6 appears to be dispersed throughout Chapter 5, and Chapter 7 of the contents page appears as Chapter 6 in the text. Had the table of contents been paginated this mistake would not have occurred.

The introduction points out that since Volumes 1 and 2 of the work appeared in 1949 and 1954, respectively, developments in the chemistry of surface-active compounds have been very rapid and some 2000 more compounds have been added to those already available commercially. From the list of abbreviations the reader can gather that the new products are spread over approximately 250 firms. These statistics, read in conjunction with the chapter on production, which includes economics, show that a distinct industry has grown up around synthetic surfactants. The fourth chapter covers in considerable detail the published and patent literature on the manufacture of these compounds.

The classification of surfactants according to constitution is one of the two main chapters of the volume and is based on the Sisley index, a very useful indication for characterising a given product briefly. The classification in the first instance is based on the ion activity, after which the three main classes are broken down according to the method of manufacture or raw materials used. The final chapter is an alphabetical index of speciality chemicals of the familiar pattern.

One would like to have seen more care taken with foreign names in a work of this international character. In addition, the book does not include products developed after 1956, which is regrettable but understandable. Nevertheless, it will certainly achieve at least the same popularity as the two previous volumes.

S. R. COCKETT

**Advanced Paint Chemistry**  
**For Students of the Paint and Printing Ink Industries**

By Phillip M. Fisk. Pp. viii + 164. London: Leonard Hill (Books) Ltd. 1961. Price, 21s. 0d.

This little book is best considered in the light of Dr. Fisk's aims, as described in the Preface, rather than the title. Believing that existing textbooks on organic chemistry and high polymers were too detailed and specialised for students following the City and Guilds Courses, Dr. Fisk wrote a series of articles for a trade journal on the organic

chemistry of those polymers and colorants of interest to the surface coatings industry. These articles are now collected together to form the basis of the book under review.

The general design is original and undoubtedly tailored to the readership in mind. In an introductory chapter on theoretical considerations, such concepts as resonance and its effect on e.g. substitution in the benzene ring, are introduced, together with an outline of the chemistry of naphthalene and anthracene, presumably intended as an introduction to pigment chemistry.

There follows a chapter on the chemistry of manufacture of some raw materials, principally petroleum chemicals, and this contains some information not usually available in textbooks of this level. The principal chapter (95 pp.), however, deals with the chemistry of high polymers, and it is on this that the value of the book will principally be judged by its users. After outlining the qualitative nature of free-radical and condensation polymerisations and the relation between molecular structure and physical properties, Dr. Fisk summarises the organic chemistry of polymers of particular interest to paint students, e.g. cellulose derivatives, aminoplasts, phenoplasts, alkyds, epoxy resins, and vinyl polymers.

In the final chapter (12 pp.) there is an extremely sketchy account of the relation between colour and constitution and of diazotisation.

The treatment is necessarily simple throughout, but is reasonably accurate. In a few cases, compression leads to errors, e.g.—(p. 79) "Crystalline substances, where regular packing occurs, will obviously be denser than substances without this orientation". (This explains why ice sinks in water!); (p. 111) "the (styrene-butadiene) surface coating emulsions remain as linear polymers . . ."; this ignores the recent introduction of stoving latices of this type which undergo vulcanisation.

Although this book will undoubtedly find a niche for itself, one is left wondering whether a student with G.C.E. "A"-level Chemistry would not do better to read the more advanced textbooks on polymers and general organic chemistry, already available, and rely on his lecturer to point out their applications to surface coatings. A more accurate title would have been "Intermediate Chemistry for Students of the Paint and Printing Ink Industries", because paint chemistry as such is not considered at any level, and I hope that Dr. Fisk will bring his undoubted talents for writing simply and attractively on technical matters to providing a truly "Advanced Paint Chemistry".

L. VALENTINE

**Manual of Man-made Fibres**

By C. Z. Carroll-Porczynski. Pp. 304. Guildford, Surrey: Astex Publishing Co. 1960. Price, 29s. 0d.

The principal section of this manual consists of tables and graphs (mainly stress-strain curves) of physical properties of fibres and flow sheets illustrating the basic steps in their production. This is followed by brief sections on textured yarns and identification; photomicrographs (85) and

X-ray diffraction photographs (31) are included. There is an extensive bibliography (39 pp.), a list of producers of man-made fibres, and finally a fairly detailed index (5 pp.).

Since the detailed information on fibre properties was provided by the manufacturers themselves, it is presumably correct and reliable. Other parts of the book, however, are not as satisfactory. In the brief section on identification (p. 177) five Tentative Textile Standards (two of which are irrelevant) and two British Standards are listed, but not BS 2792, 2793, and 2822, which deal with analysis of fibre mixtures. The bibliography, except for its subdivision by fibre type and then by manufacture, processing, etc. seems to have been compiled without rhyme or reason and is incomplete; the proof-reading also leaves much to be desired. It is not unusual for the same reference to be quoted twice within a few lines (see Turton, p. 245; Smith, p. 247; Partridge, and Patterson & Sheldon, p. 267), sometimes with minor spelling variations (see Ti(t)zka and Klop(f)stock, p. 261). None of the papers published in J.S.D.C. in 1958 on the dyeing of nylon is included in the relevant bibliography and two of those on dyeing acrylic fibres are also omitted.

Such omissions belie the claim to comprehensiveness and detract from the value of other sections, e.g. the photomicrographs and X-ray diffraction photographs. More care in both compilation and proof-reading, even if this had increased the price, would have made the book of greater value.

M.T.

#### **Problèmes Nouveaux en Teinture, Impression, Blanchiment, Apprêts**

Pp. 185. Paris: Institut Textile de France. 1960. Price, 13 NF.

This book is a collection of lectures delivered at a conference organised by the Institut Textile de France and includes eight technological papers and two of general interest. The authors are recognised experts in their own field and country and several are not unknown here.

The first paper is an essay on the French textile industry and the status of its technologists; a strongly optimistic note runs throughout, particularly with regard to the teaching of textile technology. Since the papers which follow, mostly the work of professional teachers in the industry, are of a very high standard, one can only conclude that this view is justified.

In general the papers are reviews of the present state of knowledge and are of post-graduate level. The second paper deals with the bleaching of cotton piece, both processes and machinery being covered. An excellent review of reactive dyes follows and, because of the short time-lag before publication, this includes the latest developments in a rapidly expanding field. Printing engages the attention of the author of the next paper, in which flash-ageing, the Thermosol process, and emulsion thickenings are discussed.

Continuous dyeing is treated in the fourth paper and high-temperature dyeing in the fifth. A review of fastness testing and a well-illustrated discussion of the testing of resin finishes follow. An essay on the felting of wool completes the volume.

The book is clear in print and style. It is a pleasure to read and can be recommended without reserve.

S. R. COCKETT

#### **A Statistical Manual for Chemists**

By E. L. Bauer. Pp. x + 156. New York and London: Academic Press. 1960. Price, 38s. 0d. (\$4.75).

Of the making of books on statistics there seems to be no end, and yet each has something new which makes it worth while.

The first few chapters of this book provide vivid analogies of the reasoning behind some of the usual statistical techniques. The distinction between accuracy (the correctness of a measurement) and precision (its reproducibility) is nicely drawn by an example of duck-shooting. Card-players will be amused by the list of reasonable (and unreasonable) conclusions that can be made about a batch from a sample, by the example of a few cards taken from a pack by someone who has never seen playing-cards before.

The book is particularly good on experimental design and analysis of variance, and the author obviously regards it as so important (rightly, I consider) that he introduces it as early as Chapter 3, the first two chapters being needed to expound the concepts of distributions, the average, and the standard deviation. From there, he goes on to comparison of two or more averages, correlated variables, sampling, and finally the control of routine analyses (quality control).

The Appendix of Tables is as valuable as the rest of the book. Reference is made to tables and graphs in Military Standards 105A (1950) "Sampling Procedures and Tables for Inspection by Attributes" published by the U.S. Dept. of Defense and to Military Standards 414 (1957) "Sampling Procedure and Tables for Inspection by Variables for Per Cent Defective" published by the U.S. Govt. Printing Office, Washington D.C., which might be of use to readers of the *Journal*.

It might be convenient for us all to adopt the author's abbreviations ANOVA (Analysis of Variance, p. 26), EMS (Estimated Mean Square, pp. 33-35), OC (Operating Characteristic, p. 108), and AQL (Acceptable Quality Level, p. 110), because they occur so frequently but, like AD (Average Deviation) and CL (Confidence Limits), they should be included in the list of symbols and definitions on p. vii instead of being scattered through the book on the pages indicated.

Perhaps because of his wife's criticisms—which the author says were like her coffee: hot, strong, black, and without sugar—there is little to find fault with and this short book can be safely used even by those who don't understand all of it.

L. PETERS

#### **Translation from Russian for Scientists**

By C. R. Buxton and H. Sheldon Jackson. Pp. xix + 299. London and Glasgow: Blackie and Son. 1960. Price, 30s. 0d.

This book consists mainly of a systematic treatment of Russian grammar (89 pp.), which provides what is essential for reference purposes,

together with numerous scientific sentences and texts for translation (some annotated). It is not a "teach yourself" book, for no attempt is made to guide the student systematically through the intricacies of the structure of the language. It must, in fact, be regarded primarily as a reader, and as such it is excellent. The 185 passages (136 pp.) provided for translation by students of physics, electrical engineering, chemistry, and chemical engineering are very interesting and often topical; in reading them, there will be few who will not improve their knowledge in fields other than the Russian language.

Some will not agree with the authors' view, stated in the Preface, that the Russian alphabet is a major obstacle to the student. In my experience

the alphabet is rarely a major difficulty, and I have known students who have mastered its essentials in a few hours.

No serious errors were noted, though perhaps reference to the "Law of Short Proportions" (p. 15) may puzzle chemists until they discover from the vocabulary that the replacement of two letters in the adjective alters its meaning to that of "Multiple". Another somewhat puzzling error is the expansion of the Russian abbreviation of the present title of a journal into the title which it bore some decades ago (p. 251).

As a systematic grammar and reader, the book can be recommended unreservedly to the scientist who is studying Russian.

A. E. STUBBS

#### New Books Received

*Translation from Russian for Scientists.* C. R. BUXTON and H. S. JACKSON. Pp. xix + 299. London and Glasgow: Blackie & Son Ltd. 30s. 0d.

*Index of Specifications NRC.* No. 6158. CANADIAN GOVERNMENT SPECIFICATIONS BOARD. Ottawa: Canadian Government Specifications Board. Pp. 65.

*Flotation in Paints.* I. FERGUSON. Pp. 12. British Titan Products Co. Ltd. 1959.

*Advanced Paint Chemistry.* PHILLIP M. FISK. Pp. viii + 164. London: Leonard Hill (Books) Ltd. 1961. 21s. 0d.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 879. Einsatz von Künstlichen Eiweißfasern in Mischung mit Wolle und Kaninchaar zur Herstellung von Hutfilzen. HANS GÜNTHER FRÖHLICH. Pp. 41. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 12.90.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 918. Untersuchung der Verzugsvorgänge an den Streckwerken verschiedener Spinnereimaschinen. INSTITUT FÜR TEXTILE MESSTECHNIK M. GLADBACH e.V. Pp. 43. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 13.70.

*Problèmes Nouveaux en Teinture, Impression, Blanchiment, Apprêts.* Pp. 185. Paris: Institut Textile de France. 1960. 13 NF.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 838. Zur Bestimmung der Absolutdichte von Fasern. J. JULIUS. Pp. 24. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 8.10.

*Pyridine and its Derivatives. Part 2.* ERWIN KLINGSBERG (Editor). Pp. 576. New York: Interscience Publishers Inc. 1961. 282s. 0d.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 885. Einfluss von Fettzusätzen auf das rheologische Verhalten von Schlichtefotten. INGEBORG LAMBRINOU. Pp. 57. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 16.50.

*Research Highlights of the National Bureau of Standards.* NATIONAL BUREAU OF STANDARDS. Pp. vii + 192. 1960.

*Chemie und Technologie der Silicone.* WALTER NOLL. Pp. xv + 460. Weinheim/Bergstr.: Verlag Chemie G.m.b.H. 1960. DM 39.80.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 920. Fädenpannungen beim Nassringspinnen von Bastfasern in ihrer Abhängigkeit von Fadenführung und Gestaltung von Ring und Läufer. RUDOLF OTTO and MANFRED LE CLAIRE. Pp. 54. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 16.40.

*Journal of the Rossendale Textile Society.* ROSENDALE TEXTILE SOCIETY. Pp. xxxv + 38. Rawtenstall: Rossendale College of Further Education.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 847. Untersuchungen über den Ablauf der Arbeitsvorgänge der Schlagmaschinen in Baumwoll- und Zellwollaufbereitungsanlagen. HERBERT STEIN and MARTIN EIDELBURGER. Pp. 54. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 16.70.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 846. Untersuchungen an Baumwollkarden zwecks Ermittlung der Fehlerursachen für Dickschwankungen. Pp. 46. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 14.30.

*Forschungsberichte des Landes Nordrhein-Westfalen.* No. 917. Ermittlung der Vorgänge beim Benetzen und Trocknen von Fäden unter besonderer Berücksichtigung der Arbeitsweise von Schlitzmaschinen. HERBERT STEIN and GERHARD HOISCHEN. Pp. 78. Cologne and Opladen: Westdeutscher Verlag. 1960. DM 24.10.

*Chemie und Lebenstandard.* CARL WURSTER. Pp. 18. Ludwigshafen am Rhein: Badische Anilin und Soda Fabrik. 1960.

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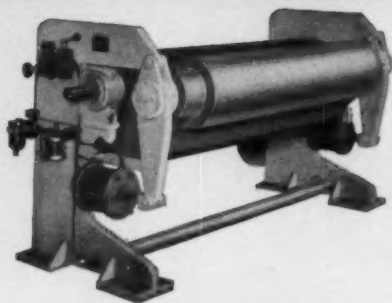
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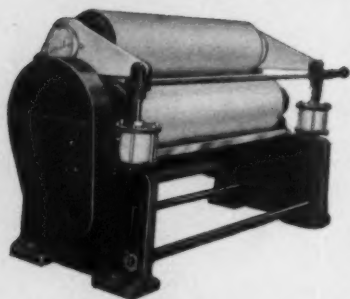
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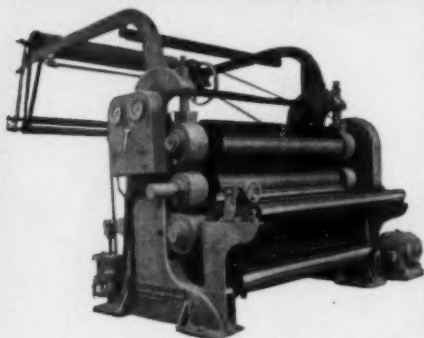
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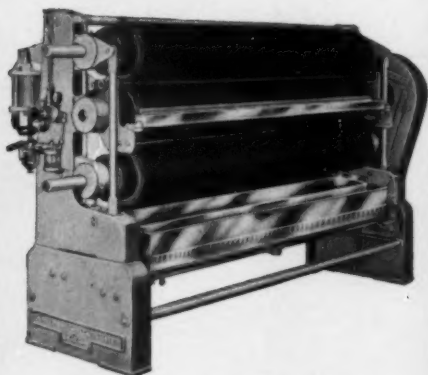
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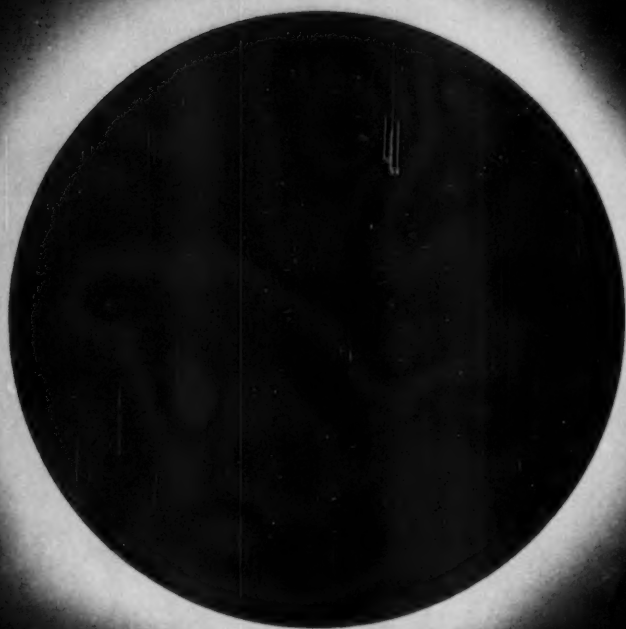


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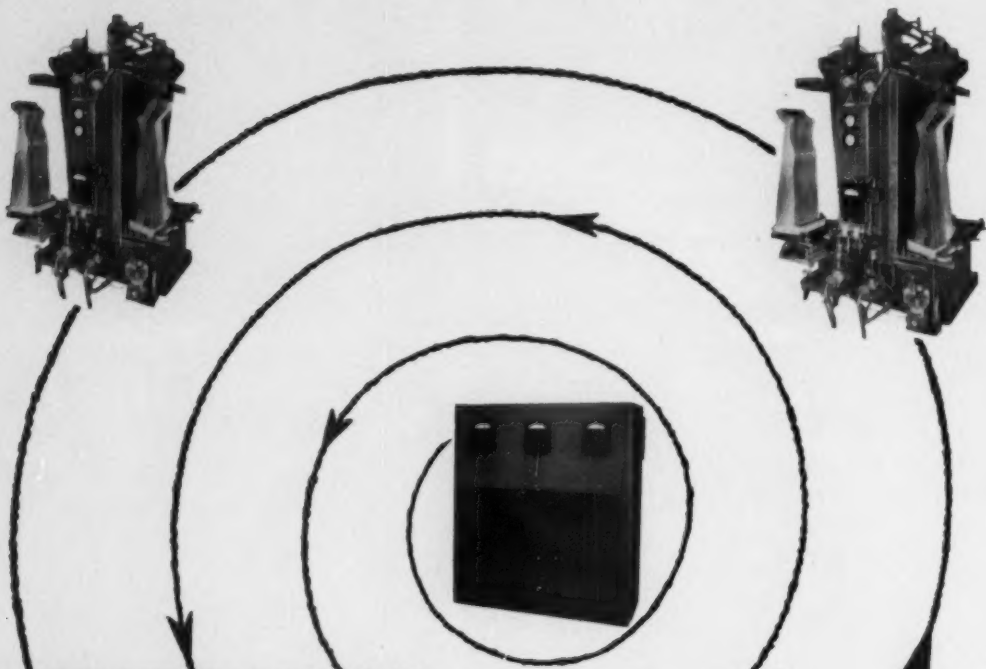
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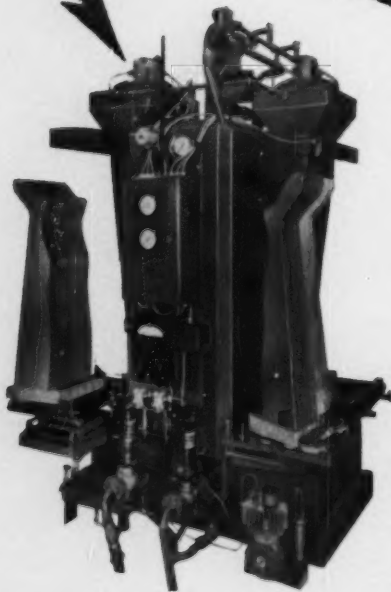


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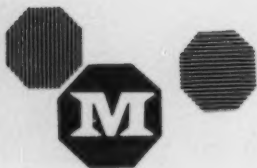
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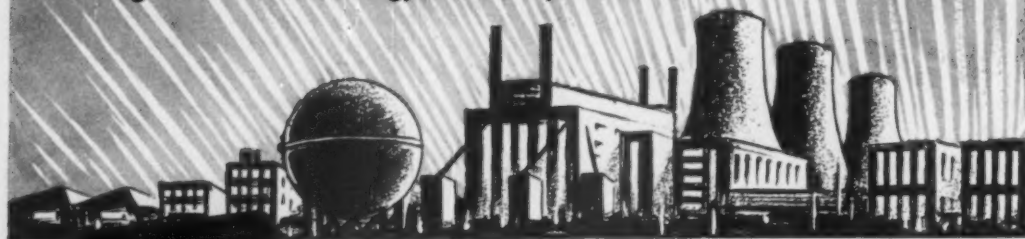
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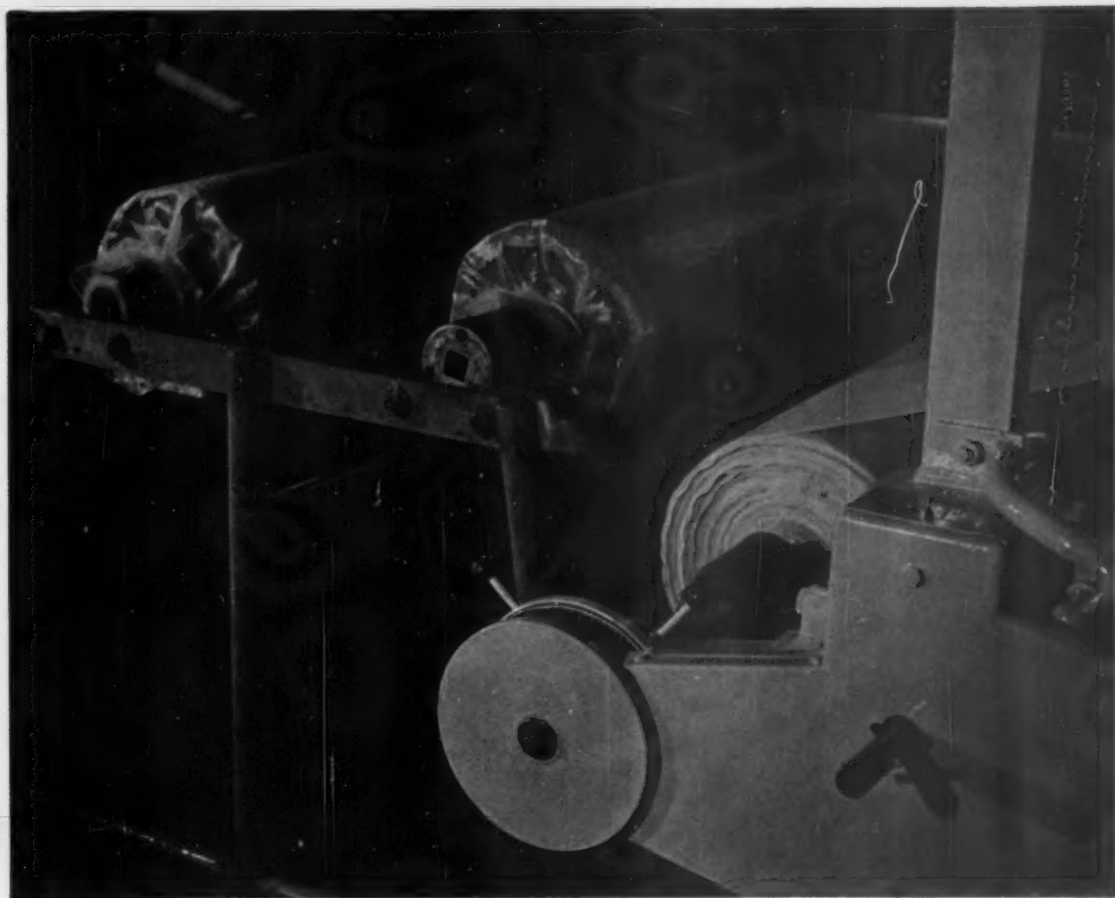
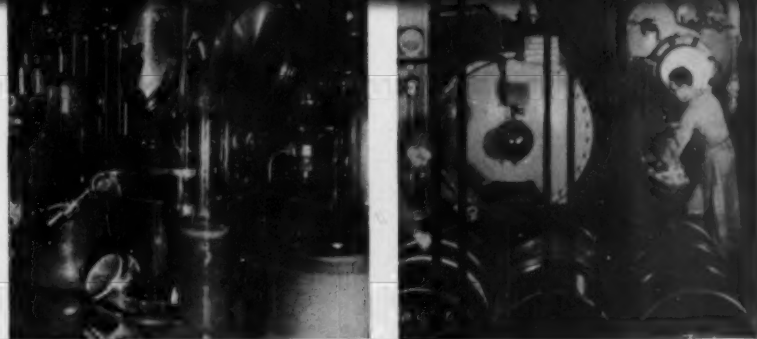
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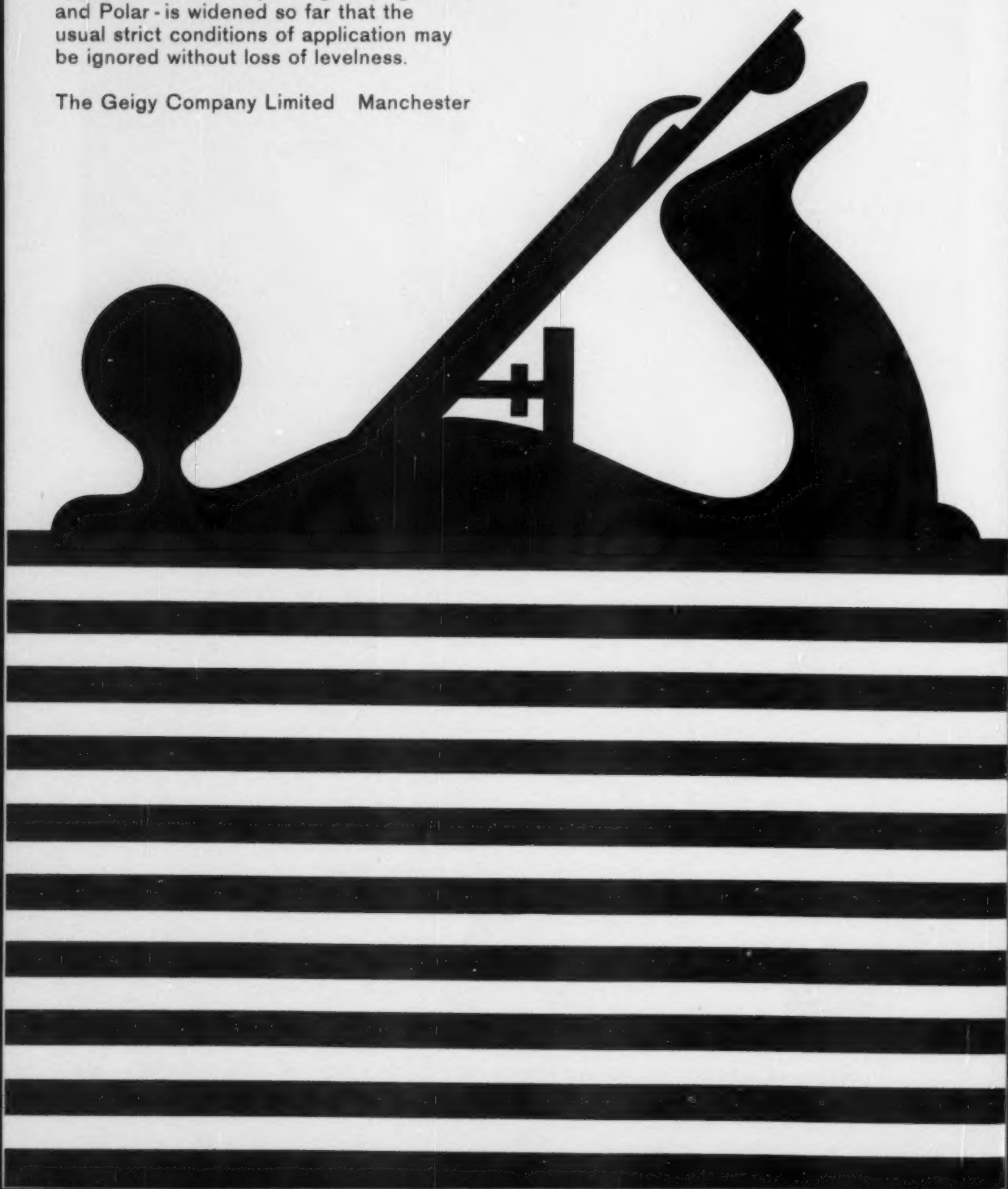
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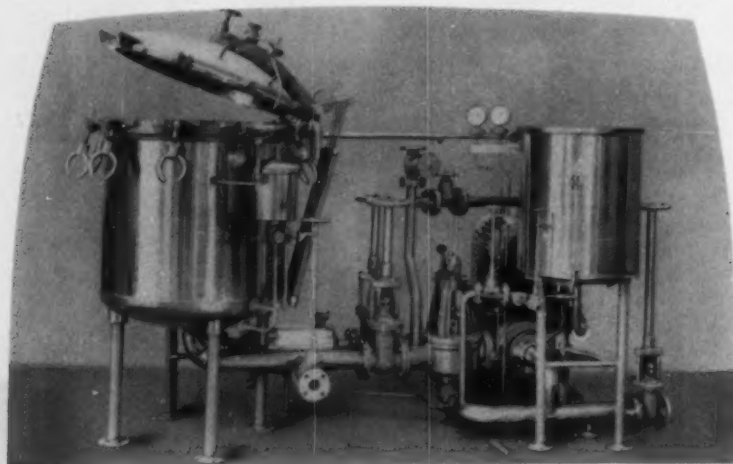
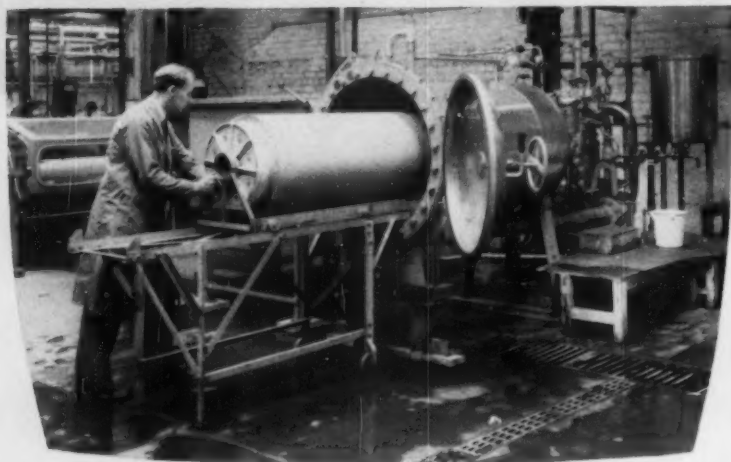
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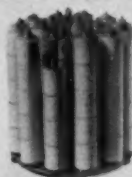
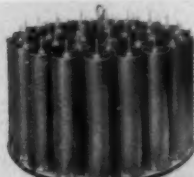
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# Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

## I—PLANT; MACHINERY; BUILDINGS

### Spray Drying of Pigment Slurries

G. Király *Brit. Chem. Eng.*, 5 (1960) 791-794  
*Chem. Abs.*, 55 (6 Feb 1961) 2209  
A proposed large-scale spray dryer for particles  $> 95 \mu$  is described. It is based on the results of experiments with a single-fluid atomiser dryer. C.O.C.

#### PATENTS

### Pelleting Lacquer

Olin Mathieson Chemical Corp.  
BP 864,274 (U.S.A. 10 Feb 1959)  
Apparatus in which a body of lacquer is simultaneously extended and cut into small pieces. C.O.C.

### Roller Felting Machine

William Bywater *BP* 859,419 (27 Feb 1958)  
Describes an improved method of mounting the adjustable rollers, improved means for adjusting these rollers and means for reciprocating the other set of rollers. C.O.C.

### Electrifier for Processing Fur and Pile Fabrics

George W. Borg Corp. *USP* 2,934,809 (3 May 1960)  
An electrifier roll has helically arranged grooves in its surface. The grooves are in sets of right and left curvatures and some of them have trailing terminal portions situated centrally of the roll, these portions being wider than the other portions of the grooves. This arrangement of the grooves avoids streakiness in the treated pile. C.O.C.

Monforts Reactor for Continuous Dyeing of Synthetic-fibre Fabrics (VIII p. 267)  
Dispersing a Colorant in Viscose (VIII p. 268)

## II—WATER AND EFFLUENTS

### Biological Oxygen Demand (B.O.D.) of Starch and Starch Derivatives

H. Benninga *Tex.*, 20 (Jan 1961) 25-28  
B.O.D. of starch products is influenced by the degree of chemical modification, dropping to one-third of the original value on slight etherification. On further etherification the B.O.D. is reduced to the same order as that of CMC and polyvinyl alcohol. R.A.

#### PATENTS

### Electrolytic Treatment of Effluents

Armour Hess Chemicals *BP* 859,417 (4 Feb 1958)  
Emulsified grease in effluents is extracted by adding an electrolyte and then treating with an electric current so as to separate the grease in a separate phase which is then isolated. C.O.C.

### Purifying Aqueous Effluents

S *BP* 863,399 (Switzerland 8 May 1958)  
Rapid and complete separation of fibre and filler material from effluents of the paper and cellulose industry is obtained by the flotation method when using as the flotation agent a mixture of sulphonated castor oil (10 parts by wt.), unsaturated carboxylic acids of 12-22 C (5-25), resinic acids (5-25), and mineral or synthetic oil (5-30), any acid groups in the components being present as alkali-metal salts. C.O.C.

## III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

### Colloids and Surface Behaviour

A. L. Draper *Ind. Eng. Chem.*, 53 (Feb 1961) 169-171  
A review of progress from late 1958 to early 1960, with 67 references. P.B.S.

### Influence of Polar Groups on the Hydrophile-Lipophile Balance of Non-ionic Compounds

N. Schönfeldt and U. Almroth  
*J.S.D.C.*, 77 (Apr 1961) 155-158  
Interactions of Surface-active Agents with Congo Red M. Hayashi

*Bull. Chem. Soc. Japan*, 34 (Jan 1961) 119-123  
The changes in the absorption spectrum of a solution of Congo Red taking place on the addition of the non-ionic

surface-active agent (Tween 80), and of the cationic agent cetyl pyridinium chloride have been studied. In the case of Tween 80, there is evidence for reaction of the surface-active agent with the dye. The absorption peak of the interaction product is at 510 m $\mu$ , irrespective of the pH of the solution, which is considered to be evidence for the formation of a molecular compound of definite constitution. As the amount of Tween 80 required to produce this spectral change increases with decreasing pH, it is concluded that interaction is dependent on the presence of Congo Red base, i.e. on availability of free amino groups. Thus the amino groups of the dye are blocked on reaction with Tween 80, the addition of which prevents aggregation of the Congo Red. Further, adsorption of the dye by filter paper or cotton is considerably reduced on addition of this agent. A shift in the wavelength of maximum absorption also takes place with cetyl pyridinium chloride. The wavelength of the new peak is 460 m $\mu$ , and this again is not affected by pH. With a ratio of 2 moles cetyl pyridinium chloride to 1 mole Congo Red, the interaction product is completely precipitated. When this molar ratio is less than 1:1, there is neither spectral shift nor precipitation, i.e. no reaction takes place, whereas in the presence of an excess of cationic agent (molar ratio ca. 4:1), the product is well dispersed and cannot be precipitated even on dilution. It is considered that Congo Red is bound ionically to cetyl pyridinium chloride at its anionic centres (the sulphonic acid groups), and that when both sulphonic acid groups are so blocked the product is most hydrophobic in character. I.G.L.

#### PATENTS

### Polyoxyethylene Ethers as Non-ionic Detergents

Atlas Powder Co. *USP* 2,934,568 (6 Feb 1950)  
Polyoxyethylene ethers of highly-branched-chain saturated acyclic alcohols of 11-15 C and in which the ethers contain on average 10-20 oxyethylene groups per mol. are better detergents than the ethers of normal aliphatic alcohols. An example is the polyoxyethylene ether of tridecyl alcohol containing 12 oxyethylene groups. C.O.C.

### Alkyl Thiosulphates as Wetting Agents

Monsanto Chemical Co. *USP* 2,892,861 (23 July 1956)  
The products obtained by treating a branched-chain alkyl halide of 12-16 C with an alkali-metal or NH<sub>3</sub> thiosulphate are excellent wetting agents. Thus 2-butyl-octyl bromide refluxed with Na thiosulphate in ethanol and water yields Na 2-butyl-octyl thiosulphate which has outstanding efficiency as a wetting agent. C.O.C.

### Starch Capable of Swelling in Cold Water

O. J. Meijer's Fabrieken voor Verpakte Artikelen  
*BP* 863,575 (Holland 31 Aug 1956)  
A product which swells in cold water is obtained by at least doubling the particle size of starch in presence of moisture by shear action at a linear pressure of 100-50,000 kg/cm and temperature below the gelatinisation point of the starch.

*BP* 863,574 (Holland 3 Oct 1956)  
Starch or a derivative thereof in which the original starch granules are still microscopically recognisable is similarly increased in particle size using a linear pressure of 100-10,000 kg/cm. C.O.C.

### Nitrogenous Derivatives of Starch having Increased Reactivity towards Formaldehyde and Formaldehyde Resins

Corn Products Co. *USP* 2,935,509 (21 Aug 1956)  
Starch carbamates are obtained by heating a mixture of starch and a compound H<sub>2</sub>N-CX-NH<sub>2</sub> (X = O or S) in presence of 5-12% of water on the weight of starch. The reaction is carried out at  $> 90^{\circ}\text{C}$  but below the decomposition temperature of the urea or other agent used. The products are thickening agents for printing pastes and in conjunction with formaldehyde or formaldehyde resin are waterproofing adhesives for textiles and paper. C.O.C.

### Dimethyl Sulphoxide as Solvent for Polyhydroxylic Compounds and its Use as Reaction Medium in making Polyether Derivatives of Polyhydroxylic Compounds

ICI BP 864,097 (26 Feb 1958)  
Dimethyl sulphoxide is an excellent solvent for many polyhydroxylic compounds, does not react with alkylene oxides or other epoxides, and is not decomposed by the alkaline catalysts used in formation of alkylene oxide or epoxide addition compounds. Furthermore it can be recovered from the reaction mixture by distillation under reduced pressure. Thus polyhydroxylic compounds of m.p. > 120°C are dissolved in dimethyl sulphoxide and treated with alkylene oxides and/or polyepoxides to yield the polyether derivatives of the polyhydroxylic compounds.

C.O.C.

### Carriers for Dyeing or Printing Polyester Fibres with Disperse Dyes

FH BP 860,441 (Germany 28 Apr 1956)  
Compounds containing at least one lactone group and one aromatic nucleus and which are insoluble or sparingly soluble in water, e.g. the lactone of 2-hydroxynaphthalene-1-β-propionic acid, are excellent carriers when disperse dyeing at 97–98°C. Alternatively, a hydroxy acid may be used whose lactone ring is closed during dyeing. The carrier may be either used in the dyebath or applied to the polyester fibres before they enter the dyebath.

C.O.C.

### Stable Aqueous Resin Emulsions

Ciba BP 859,709 (Switzerland 6 Mar 1957)  
Stable oil-in-water emulsions of resins are obtained by removing the alcohol from a lacquer composed of (1) a hardenable water-insoluble etherified methylol compound of amide character, e.g. a melamine-formaldehyde condensate etherified with n-butanol, and (2) an alcohol of 3–7 C. The alcohol is extracted from the lacquer by a liquid which does not dissolve the resin. The separated etherified methylol compound is then emulsified in water in presence of an emulsifying agent.

C.O.C.

### Water-repellent and Rot-proofing Composition

Chemische Werke Witten BP 855,718 (Germany 23 Dec 1957)  
Textiles are rendered highly water-repellent and rot-proof by treatment with a water-repellent agent and relatively small amounts of chlorinated phenol esters and chlorinated phenols. Thus granulated size (6-80) was dissolved in water (18-85), urea (0-20) added and the solution heated for 30 min. Paraffin wax (14-00), montan wax (2-10), pentachlorophenol esters of C<sub>6</sub>-C<sub>13</sub> acids (8-25), and pentachlorophenol (2-50) were added at 70°C followed by 18% aq. Al trifluoride (4-50) and 30% acetic acid (2-30). The concentrate was diluted with 4 times its weight of water. Cotton cloth impregnated with its own weight of the diluted concentrate and dried at 80°C had high water repellency and good durability in a soil-burial test.

C.O.C.

### Sulphonated Urea-Thiourea-Formaldehyde Resins—Flame-resisting Agents

American Cyanamid Co. BP 856,466 (U.S.A. 30 Apr 1956)  
Urea (1 mol.) is treated at > 40°C and pH 7–9 with a water-soluble aldehyde (2-0-6-5 mol.) in presence of bisulphite (0-01-0-040 mol. per mol. aldehyde). The product is polymerised at pH 2-2-3-8 and > 40°C, the mixture brought to pH 7-9 and thiourea added (0-3-2-5 mol.). The pH is then brought to 3-5-5-5 and the product alkylated with a saturated aliphatic alcohol at 1-3 C (0-3-4-0 mol.) which may have been added at any time during the previous reaction sequence. The mass is then rendered alkaline to yield a hydrophilic resinous product which neither flocculates when kept for 2 weeks at 25°C nor precipitates when diluted with 50 times its volume of water at 25°C. The product is used for imparting a flame-resistant finish to textiles, particularly to nylon netting.

C.O.C.

### Vinyl Phosphoric Acid and its Derivatives—Flame-resisting Agents

FH BP 858,057 (Germany 14 Mar 1956)  
Vinyl phosphoric acid dichloride CH<sub>2</sub>:CH·POCl<sub>2</sub>Cl is obtained by treating a vinyl phosphoric diester with PCl<sub>5</sub>. Both this compound and its derivatives, e.g. vinylphosphonic acid, obtained by treating the dichloride with water in an inert organic solvent, are flame-resisting

agents. Thus, impregnating a cellulosic textile with a 10–20% solution of vinyl phosphonic acid dichloride in an inert volatile organic solvent, treating with ammonia and finally baking at 130–150°C, imparts a good flame-resisting finish.

C.O.C.

### Colour Stabilisation of Acrylonitrile Solutions

American Cyanamid Co. BP 859,262 (U.S.A. 1 Nov 1956)  
Aqueous solutions of acrylonitrile are effectively stabilised by incorporation of 0-1-5-0% (on dry weight of the acrylonitrile) of a water-soluble inorganic bisulphite.

C.O.C.

### Keratinolytic Enzymes

W. Y. Koh and R. A. Messing BP 860,312 (U.S.A. 25 Nov 1957)  
Keratinases can be produced by cultivating under aerobic conditions *Aspergillus flavus*: ATCC No. 13003 and *A. niger*: ATCC No. 13004.

C.O.C.

### Solutions of Azo Pigments (IV p. 260)

Tanning Agents from Ammonium Bisulphite and Phenol-Formaldehyde Condensates (XII p. 272)

Siloxane Compositions for rendering Leather Water-repellent (XII p. 272)

Ultraviolet Absorbers in Plastics (XIII p. 273)

## IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

### Salts of Naphthalene-1- and -2-Sulphonic Acids. Solubility Diagram of the Quaternary System Na<sup>+</sup>-K<sup>+</sup>-C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>(1)<sup>-</sup>—C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>(2)<sup>-</sup>—H<sub>2</sub>O

R. Flatt and A. Villard  
*Helv. chim. Acta*, 44 (Feb 1961) 148-158

A method is given for the determination of the anion C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>(2)<sup>-</sup> (anion B<sup>-</sup>) in presence of C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>(1)<sup>-</sup> (anion A<sup>-</sup>) to an accuracy of 1-2%. The existence of the double salt, C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>(1)K, C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>(2)K, 1H<sub>2</sub>O, is established and its preparation described. The sol. diagrams of the quaternary system Na<sup>+</sup>-K<sup>+</sup>-A<sup>-</sup>-B<sup>-</sup>-H<sub>2</sub>O at 0, 25, and 50°C, are constructed from 254 sol. analyses.

H.H.H.

### Copper Complexes with some Formazans Derived from o-, m-, and p-Aminobenzoic Acids

M. Petrovanu and A. Borcea  
*Analele stiint. univ. "Al. I. Cuza" Iasi, Sect. I. [N.S.]*, 4 (1958) 157-168

*Chem. Abs.*, 55 (9 Jan 1961) 432

Diazotised o-, m-, and p-aminobenzoic acids were coupled with phenylhydrazones of fatty, unsaturated, and aromatic aldehydes (e.g. enanthic, anisic, and cinnamic aldehydes) to give formazans, which gave 1:1 Cu-complexes with aq. copper acetate. Thus N-(carboxy-3-phenyl)-N'-phenyl-C-isobutylformazan gave a bright red Cu-complex.

E.S.

### Chromoisomerism of p-Nitrobenzyl Cyanide

F. Nerdel, D. Klamann, and W. Ebing  
*Ann.*, 632 (1960) 55-65

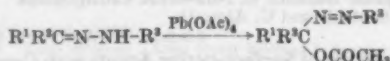
*Chem. Abs.*, 55 (9 Jan 1961) 443

The suggested chromoisomerism of the Na salt of p-nitrobenzyl cyanide (I) was shown not to exist and the colour changes found in polar solvents are explained by intermol.-intramol. oxidation-reductions, giving a series of dyes. By, e.g., reprecipitation of I from HCl and subsequent chromatography, a series of coloured products was obtained. Details are given for the preparation of I and of numerous related and derived compounds. A.T.P.

### Preparation and Structure of Azocetates, a New Class of Compound

D. C. Iffland, L. Salisbury, and W. R. Schafer  
*J. Amer. Chem. Soc.*, 83 (5 Feb 1961) 747-749

Lead tetra-acetate has been shown to react with keto-hydrazones in methylene chloride at 0-10°C to form azocetates according to the scheme



where R<sup>1</sup> = methyl, phenyl or is conjoined with R<sup>2</sup> to give a cyclic -(CH<sub>2</sub>)<sub>n</sub>- structure; R<sup>3</sup> may also be methyl, ethyl, or phenyl, R<sup>3</sup> = methyl, phenyl, para-substituted

or 2,4-disubstituted phenyl residues. Generally high yields of easily isolated products were obtained and their structure was established from analysis of u.v. spectra. Azooacetates prepared from arylhydrazones gave absorption spectra similar to those of the alkylazobenzenes. A radical mechanism for the reaction is proposed and the chemical properties of these opd. are currently being investigated.

F.J.

### Coal-tar Dyes. XXIV—FD&C Red No. 4 (C.I. Food Red 1)

J. A. Wenninger, J. H. Jones, and M. Dolinsky  
*J. Assocn. Offic. Agric. Chem.*, **43** (1960) 805-809  
*Chem. Abs.*, **55** (20 Feb 1961) 3983

The isomers of FD&C Red No. 4, 2-(3-sulpho-2,5-xylylazo)-1-naphthol-4-sulphonic acid and 2-(4-sulpho-2,5-xylylazo)-1-naphthol sulphonic acid, were prepared. Methods for separating them chromatographically from FD&C Red No. 4 were worked out. Commercial samples of the dye contained  $> 0.5\%$  of the first and  $< 5.0\%$  of the second isomer. C.O.C.

### Investigations in the Anthraquinone Series. XXXI—Sulphonic Acids of *trans*-Dibenzopyrenequinone

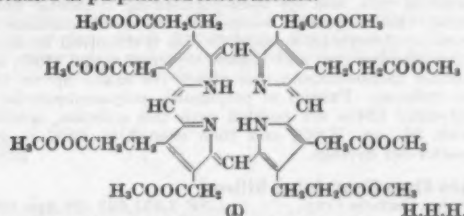
V. V. Kozlov and E. A. Silaeva  
*Zhur. obshch. khim.*, **30** (Nov 1960) 3766-3772

Sulphonation of *trans*-dibenzopyrenequinone (I) yields a disulphonic acid to which the  $\beta, \beta'$  structure is assigned, since when the sulphonate groups are replaced by bromine there results  $\beta, \beta'$ -dibromo-*trans*-dibenzopyrenequinone. Detailed experimental directions are given. G.J.K.

### A Pigment of Pearl-oyster Shells

Y. Iwakiri, M. Yamaguchi, and T. Tsumaki  
*Mem. Faculty Sci., Kyushu Univ.*, **3** (Dec 1960) 161-164

Pigment was extracted by a combination of the methods of Takagi and Tanaka (*J. Chem. Soc. Japan, Pure Chem. Sect.*, **76** (1954) 406), and of Sawada (*Bull. Natl. Pearl Res. Lab.*, **4** (1958) 335), and its constitution is now confirmed as uroporphyrine I octamethylster (I). The Cu salt was obtained as purplish red felted needles.



### Carotene Pigments in Marigold Petals

S. D. Morchan  
*Zhur. priklad. khim.*, **33** (Feb 1960) 484-486  
Four pigments were isolated in cryst. form from petals of *Calendula officinalis* and identified as  $\beta$ -carotene, lycopene, violaxanthin, and rubixanthin. In addition a paraffin hydrocarbon hentriacontane of formula  $C_{31}H_{64}$ , and two phytosterols were also found. T.Z.W.

### Increasing the Fastness of Paris Blue (C.I. Pigment Blue 27) to Alkali

G. Szigeti and A. Erdélyi  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 317-321  
*Chem. Abs.*, **55** (6 Feb 1961) 3087

$FeSO_4$  and  $K_4[Fe(CN)_6]$  were mixed together in presence of Ni ions to yield a white sludge  $K_2(Fe_3Ni_2)[Fe(CN)_6]_4 \cdot nFeSO_4$ . The value of  $y$  was a function of that of  $z$ . Increasing Ni content (incorporated into the molecule rather than adsorbed) increased the fastness to alkali but deteriorated the colour. Boiling the sludge in acid did not increase the fastness to alkali. The amount of Ni taken up was unaffected by changes in the  $FeSO_4:K_4[Fe(CN)_6]$  molecular ratio, nor did it change during oxidation. C.O.C.

### Studies of Inorganic Pigments at the Chemical Industry University, Veszprém, Hungary

K. Polinszky  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 253-259  
*Chem. Abs.*, **55** (6 Feb 1961) 3087

Reviews work carried out on Black, Red, and Yellow Iron Oxides (C.I. 77491, 77492, 77499), Yellow ZnO, Red Cr oxide, and Prussian Blue-type pigments. C.O.C.

### Manufacture of Aluminium Hydroxide (C.I. 77002) as a Pigment for Printing Ink

L. Somkuti  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 273-279  
*Chem. Abs.*, **55** (6 Feb 1961) 3087

Study of the manufacture of soft and loose  $Al(OH)_3$  suitable for use in printing inks. When precipitating with  $Na_2CO_3$ , increase in temperature of precipitation and/or sludge concentration increased the density of the product, pH had no effect on pigment density, increase in temperature of precipitation increased the rate of precipitation. The softest pigment was obtained by drying at  $< 60^\circ C$ . Pigments produced by precipitating with  $NH_4OH$  were suitable only as fillers for rubber. C.O.C.

### Loss of Chromium in the Manufacture of Green Chromium Oxide (C.I. 77288)

F. Henszelmann  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 295-302  
*Chem. Abs.*, **55** (6 Feb 1961) 3087

When the  $K_2Cr_2O_7:S$  ratio was reduced to below 100:15 in manufacture of green  $Cr_2O_3$  by reduction with  $S$ , the loss of  $Cr$  increased, the pigment became difficult to grind because of the increased density, and the hue and colour strengths deteriorated. Decreasing the particle size of the raw materials to an average of 0.2 mm led to decrease in loss of  $Cr$ ; further grinding gave no further improvement. Addition of  $Na_2Cr_2O_7$  to the  $K$  salt lowered the reaction temperature and thus caused increased loss of  $Cr$ . The apparent reduction in loss of  $Cr$ , determined iodometrically, when part of the  $S$  was replaced by charcoal and/or pine rosin, was probably caused by the effect of these replacements on the analyses rather than the actual reduction in loss of  $Cr$ . C.O.C.

### High-pressure Manufacture of Green Hydrated Chromium Oxide (C.I. 77289)

F. Henszelmann and L. Zalka  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 303-309  
*Chem. Abs.*, **55** (6 Feb 1961) 3087

Pigments,  $Cr_2O_3 \cdot nH_2O$  ( $n = 1, 2$  or  $3$ ), were prepared by reducing  $K$  or  $Na$  chromate solutions under 101 atm. pressure at  $310^\circ C$  for 3 h. Catalysts were unnecessary. The resulting pigments had good colour value and covering power. C.O.C.

### Manufacture of Red Iron Oxide by Calcining Ferrous Sulphate (C.I. 77491)

G. Sziklai  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 267-272  
*Chem. Abs.*, **55** (6 Feb 1961) 3087

$FeSO_4 \cdot H_2O$  was made by mixing  $FeSO_4 \cdot 7H_2O$  with  $FeSO_4$  and drying at  $100^\circ C$ . The best pigments were produced by calcining the  $FeSO_4 \cdot H_2O$  at  $600-650^\circ C$ ; at lower temperatures the products had too high a sulphate content, and at higher temperatures they were too blue. At  $650^\circ C$  the reaction velocity was twice that at  $600^\circ C$ . After calcining for 3 h at  $650^\circ C$  the product contained 4.55% sulphate. To bring this to below the permissible maximum of 1.5% the pigment was drowned in cold water immediately on leaving the furnace, rinsed by sedimentation and decanting, and dried. The product contained  $Fe_2O_3$  98.56,  $FeO$  0.48, and  $SO_3$  0.98%. C.O.C.

### Manufacture of Black Iron Oxide (C.I. 77499) in Presence of Nitrates

G. Szigeti  
*Veszprémi Vegyipari Egyetem Közleményei*, **2** (1958) 261-266  
*Chem. Abs.*, **55** (6 Feb 1961) 3088

$Ca(OH)_2$  suspension was stirred into  $FeCl_3$  suspension at  $90-92^\circ C$  and pH 6-7, stirring continued for 5 min, aq. nitrate added, and then air bubbled through until the black pigment was fully developed. After precipitation of  $Fe(OH)_2$  was completed, there were only traces of bivalent  $Fe$  in the mother liquor; by increasing the degree of oxidation this increased to a maximum  $Fe(III):Fe(II)$  ratio of 1.9-2.0 and decreased thereafter. Varying amounts of  $FeCl_3$  were adsorbed on the surface of the precipitated  $Fe(OH)_2$ .  $K$ ,  $Cu$ , and  $Na$  nitrates were equally suitable and bubbling through of excess of air had no further effect. Oxidation rate was not affected by the pigment concentration in the mixture. The range of bivalent  $Fe$  excess at which stable pigments could be prepared was 3-4%.

Addition of nitrates reduced the time needed for oxidation. Using nitrates in amounts ensuring excess of bivalent Fe over trivalent Fe in the pigment resulted in a considerably improved product. C.O.C.

#### Effects of Manufacturing Conditions and Chemical Composition on the Hue of Basic Lead Chromate (C.I. 77601)

J. Scholtz *Veszprémi Vegyipari Egyetem Közleményei*, 2 (1958) 281-286

*Chem. Abs.*, 55 (6 Feb 1961) 3087

Red pigments of good covering power and fastness were obtained by dissolving PbO in a 90:10 mole mixture of HCl and acetic acid at 80-85°C and precipitating at that temperature and pH 7 with dichromate to yield the products containing PbO and PbCrO<sub>4</sub>. Excess of PbO in the products acted only as diluent and did not add to the colour value. C.O.C.

#### Manufacture of Zinc Chromate (C.I. 77955)

J. de Jonge *Veszprémi Vegyipari Egyetem Közleményei*, 2 (1958) 287-293

*Chem. Abs.*, 55 (6 Feb 1961) 3088

Pigments of composition 4ZnO·CrO<sub>3</sub>·3H<sub>2</sub>O to 4ZnO·4CrO<sub>3</sub>·K<sub>2</sub>O·3H<sub>2</sub>O were prepared. Complete removal of contaminating sulphate was found to be impractical. The sulphate content could be minimised by reducing the amount of H<sub>2</sub>SO<sub>4</sub> and increasing the amount of alkali dichromate used, by using solutions as dilute as possible and by washing the pigments by alternating sedimentation and decanting. Decrease of sulphate content on storage was negligible. One mole ZnO can hold a maximum of 0.5 mole K<sub>2</sub>CrO<sub>4</sub>, and any excess of the latter was lost when filtering. To keep loss of Cr to a minimum, exactly 0.25 mole H<sub>2</sub>SO<sub>4</sub> per mole ZnO should be used. C.O.C.

#### PATENTS

##### 5-Pyrazolone Colour Couplers

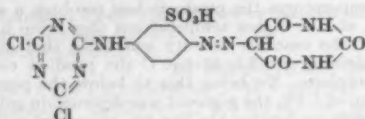
Eastman Kodak Co *USP* 2,933,391 (6 Sept 1956)

1-Phenyl-3-alkyl-5-pyrazolone colour couplers having < 2 Hal substituted in the phenyl radical and Alk of 11-17 C, e.g. 1-(2,4,6-trichlorophenyl)-3-pentadecyl-5-pyrazolone, are stable to light and heat when in photographic emulsions. They yield magenta dyes whose secondary light absorptions are at a minimum. C.O.C.

##### Monoazo Compounds having a Barbituric Acid Residue—Reactive Dyes for Cellulose

Ciba *BP* 863,155 (Switzerland 3 Aug 1956)

Condensates of 1 mol. of cyanuric chloride with 1 mol. of a diaminobenzenesulphonic acid are diazotised and coupled with barbituric acid to give the title dyes; or cyanuric chloride may be condensed with a suitable aminobenzenesulphonic acid compound. Thus the condensate of 1 mol. of cyanuric chloride with 1 mol. of 1,4-diaminobenzene-3-sulphonic acid, diazotised and coupled with barbituric acid, gives—

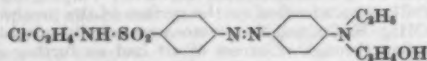


which dyes cellulose greenish yellow from an alkaline bath containing salt at 25-35°C. E.S.

##### Monoazo Disperse Reactive Dyes

ICI *BP* 862,269 (4 Oct 1957)

Monoazo disperse dyes containing a group -SO<sub>2</sub>NR<sup>1</sup>R<sup>2</sup> (R<sup>1</sup> = chloro- or bromo-alkyl or chloro- or bromo-cycloalkyl; R<sup>2</sup> = H or a subst. hydrocarbon radical, or chloro- or bromo-alkyl or chloro- or bromo-cycloalkyl, or R<sup>1</sup> and R<sup>2</sup> with the N atom form a ring containing Cl or Br) react with nylon under alkaline conditions to give dyeings of good wet fastness, and may be applied also to other synthetic fibres, e.g. cellulose acetate and polyester fibres. Thus diazotised sulphanil(β-chloroethyl)amide coupled with N-ethyl-N-β-hydroxyethyl-aniline gives—



which dyes nylon orange of good wet fastness when aftertreated in hot aq. 0.2% Na<sub>2</sub>CO<sub>3</sub>. E.S.

##### Preparation of Azo Pigments in Absence of Water

Ciba *BP* 861,443 (Switzerland 7 Mar 1956)

Azo pigments free of SO<sub>2</sub>H groups may be prepared in absence of water by kneading a stabilised diazo compound with a coupling component in presence of a small amount of an organic liquid and an acid-binding agent if necessary. The method is particularly useful when disazo pigments are required from coupling components which do not readily couple twice under normal aqueous conditions. Pigments produced in this way have sometimes to be aftertreated in a solvent to get them into a useful physical form. Thus a small kneading apparatus is charged with 3,3'-dichloro-4,4'-bis(3'-hydroxy-2'-naphthylamino)diphenyl (0.05 g mol.) and pyridine (20 ml) to produce a plastic mass. Whilst cooling, there is sprinkled in the ZnCl<sub>2</sub> double salt of m-chlorodiazobenzene chloride (0.1 g mol.) and further pyridine (40 ml) to maintain a plastic consistency. The α-form of the disazo pigment so formed is transformed into the desired β-form by boiling in o-dichlorobenzene for 5 min before cooling and filtering off. E.S.

##### Solutions of Azo Pigments

Ciba *BP* 862,565 (Switzerland 5 Mar 1956)

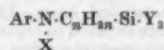
Azo pigments insoluble in water or aq. alkaline solutions and at the most slightly soluble in organic solvents, can be dissolved in a mixture of a strongly basic substance and a weakly to moderately polar liquid of dielectric constant < 6 and of acidity less than that of ethanol. The pigment should contain a very weakly acid group (preferably OH ortho to an azo link). Suitable strong bases include alkali-metal hydroxides, ammonia and saturated cyclic amines, and for the weakly to moderately polar liquid, ethylene glycol and polyethylene glycol as well as liquid amines, e.g. aniline and ethanalamines, are suitable.

The solutions so formed may be used for dyeing or printing, the fibre being impregnated with the solution and the insoluble pigment subsequently precipitated in the fibre by dilution with water or by heat. They may also be used for dyeing anodised aluminium, for colouring lacquer and printing inks, and for mass-pigmenting synthetic fibres. Thus the monoazo compound 2,5-dichloroanilino-3-hydroxy-2-naphtho-o-aniside (14) is dissolved in monoethanolamine (50) and hexamethylenediamine (100), and further monoethanolamine added (to make up to 1000 by volume). Fabrics of polyamide, polyacrylonitrile, or polyester fibres are padded with this solution, acidified with 5% aq. H<sub>2</sub>SO<sub>4</sub> and then soaped at 95°C to give scarlet-red dyeings. E.S.

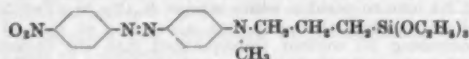
##### Azo Dyes Containing Silicon

Union Carbide Corp. *USP* 2,931,693 (29 Apr 1957)

Diazo and tetrazo compounds couple with arylamino-alkylsilanes

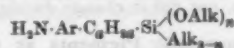


(X = H or Alk; Y = Alk, Ar, OAlk, or OAr; n = integer, preferably 3, 4 or 5), to give mono- and dis-azo dyes or pigments, with properties depending mainly upon the diazo or tetrazo compound used. Thus a soln. of p-nitroaniline in dimethylformamide containing HCl and acetic acid was diazotised by addition of NaNO<sub>2</sub> dissolved in ethylene glycol. Coupling in dioxan with N-methyl-N-(γ-triethoxysilylpropyl)aniline gave a deep red viscous liquid which gradually solidified giving crystals of

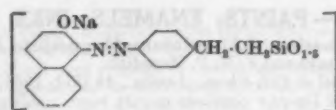


*USP* 2,927,839 (29 Apr 1957)

Aminoaryloxyalkoxysilanes



(n = integer, preferably 0-4; n = 1, 2 or 3) diazotised and coupled with suitable coupling components give azo dyes of various types, depending on the nature of the coupling component used. Thus diazotised 4-(β-triethoxysilyl)ethylaniline coupled with β-naphthol gives a red compound containing the unit



E.S.

**Sulphur Dyes**

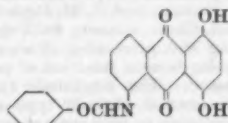
CFM

BP 862,218 (Germany 11 June 1958)  
Bright orange to yellow-brown sulphur dyes are obtained by brominating decacyclic and treating the products (especially weakly brominated ones, e.g. tribromide) with S at < 195°C. They have good affinity and fastness and are faster to light than the sulphur dyes of similar colour obtained from *m*-tolylenediamine (cf. BP 765,636). A.T.P.

**Disperse Dyes for Polyesters—1,4-Dihydroxy-5-Acylaminoanthraquinones**

Ciba

BP 862,234 (Switzerland 27 May 1957)  
Acylation of 5-aminoquinizarin (I) with halides of low-mol.-wt. monocarboxylic acids (especially of the benzene series) gives orange-red dyes of good light and sublimation fastness. Dyes of this colour and fastness were hitherto unknown. Thus, benzoyl chloride (1.8) is added to I (2.55) in dry monochlorobenzene (40) and the mixture refluxed for 2 h, cooled to 5°C and filtered to give



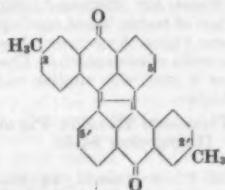
an orange dye of excellent fastness.

A.T.P.

**Acedianthrone Vat Dyes**

Ciba

BP 861,758 (Switzerland 4 Oct 1956)  
Monobromoacedianthrone is condensed with a mono- or di-aminoanthraquinone and dibromoacedianthrone with a monoaminoanthraquinone (in such proportions that one Br atom reacts with one NH<sub>2</sub> group), and the resulting anthrimides optionally carbonylated to give strongly dyeing brown vat dyes which do not cause fibre damage. Thus, nitrobenzene (50), monobromo-2,2'-dimethylacedianthrone (3.5) obtained by bromination of



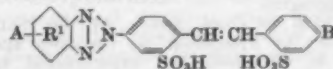
in ClSO<sub>3</sub>H, 1-aminoanthraquinone (2), Cu<sub>2</sub>Cl<sub>2</sub> (0.4), Cu powder (0.2) and Na<sub>2</sub>CO<sub>3</sub> (0.5) are heated at 210–220°C for 20 h. The product is filtered at 100°C, washed with nitrobenzene and alcohol, and extracted with dilute HCl to give a strong reddish brown vat dye.

A.T.P.

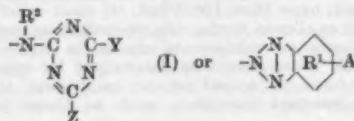
**Water-soluble Stilbene Reactive Dyes**

ICI

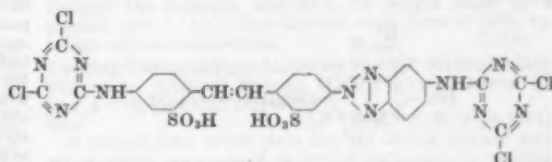
BP 862,225 (29 Sept 1958)  
The above have formula—



(R<sup>1</sup> = subst. or unsubst. benzene, or part of naphthalene nucleus, B = Cl, Br, NO<sub>2</sub>, acylamino, NN-dialkyl-amino,



A = H or (I); R<sup>1</sup> = H or Alk of 1–4 C, Y = Cl or Br; Z = Cl, Br, Alk, Ar, Alkoxy, Aryloxy, thioaryl, thioalkyl, NH<sub>2</sub> or subst. amino; the dye contains at least one triazinylamino group). They are reactive dyes for cellulose, wool, and polyamides and, when applied with an acid-binding agent, yield greenish yellows of excellent fastness to light and washing. Thus—



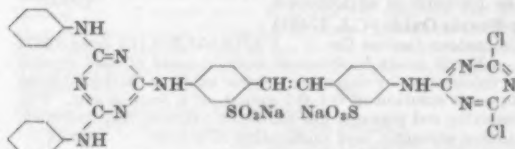
is obtained by condensing 1 mol. appropriate diamine and 2 mol. cyanuric chloride.

A.T.P.

**Fluorescent Brightening Agent for Cellulose**

Ciba

BP 864,900 (Switzerland 8 Aug 1956)  
Fluorescent brightening agents containing at least one triazine ring substituted by 2 halogen atoms or one halogen atom and either a hydroxy or alkoxy group of < 4 C are fixed on the cellulose with aid of an agent capable of binding hydrogen halide. Thus cotton yarn is treated for 20 min at room temperature in an aqueous solution containing



and Na<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>CO<sub>3</sub> is then added, the bath raised to 50°C and treatment continued for 30 min.

C.O.C.

**Organic-solvent-soluble Fluorescent Brightening Agents**

Gy

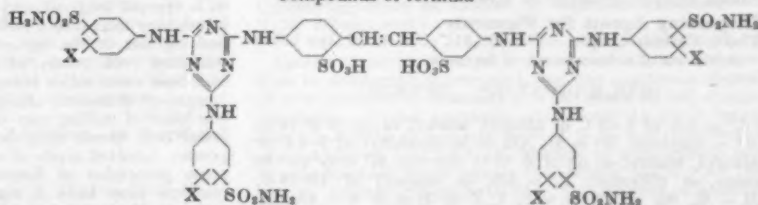
BP 863,164 (Switzerland 6 Sept 1956)  
4,5-Arylene-1,2,3-triazolyl cyanostilbene compounds containing no acid- or salt-forming groups or colouring substituents and which are soluble in organic solvents are fluorescent brightening agents for use in hydrophobic organic materials, e.g. waxes, oils, polyethylene, polyacrylonitrile, etc. Thus 2-[2'-cyanostilbyl-4']-(acenaphtheno-4',5':4,5)-1,2,3-triazole is an excellent fluorescent brightening agent for nylon and polyester fibres.

C.O.C.

**Bis-triazinylaminostilbenedisulphonic Acid Derivatives—Fluorescent Brightening Agents**

Société de Produits Chimiques et de Synthèse

BP 862,851 (France 31 May 1958)  
Compounds of formula—



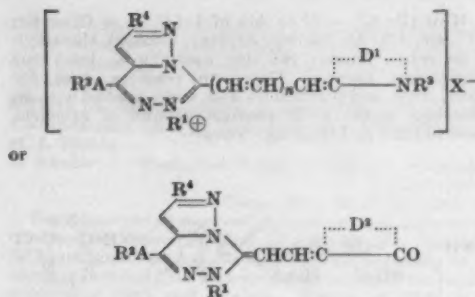
(X = H, Hal or CH<sub>3</sub>) have low solubility in water but good affinity for wool and nylon as well as for cellulose. They are prepared by treating 4,4'-diaminostilbene-2,2'-disulphonic acid (1 mol.) first with cyanuric chloride (2 mol.) and then with an amino benzenesulphonamide (4 mol.). An example is bis[2,4-di-*p*-sulphonamidophenyl-amino-1,3,5-triazyl-(6)]-4,4'-diaminostilbene-2,2'-disulphonic acid, which has low solubility in water and strong blue fluorescence. It imparts an excellent white to cotton and nylon from neutral medium and to wool from an acid bath.

C.O.C.

**Cyanine and meroCyanine Dyes**

Ilford

Dyes of formula—  
BP 862,825 (6 Feb 1957)



(R<sup>1</sup>, R<sup>2</sup>, and R<sup>3</sup> = Alk or aralkyl; R<sup>4</sup> = Alk or Ar; A = O or S; n = 0 or 1; D<sup>1</sup> = atoms to complete a 5- or 6-membered ring; D<sup>2</sup> = atoms to complete a 5- or 6-membered keto-methylene nucleus, e.g. the yellow 3-ethyl-5-(6,7-dihydro-6-methyl-4-methylthio-2-phenyl-1,5,6-triazaindolizinyldene-7-ethylidene)-2-thio-4-thiazolidene, have photosensitising properties. C.O.C.

#### Calcium Carbonate (C.I. Pigment White 18)

Wyandotte Chemicals Corpn. BP 863,864 (11 Apr 1958)

CaCO<sub>3</sub> of particle size > 0.1 μ is treated with a primary n-alkylamine of 10-18 C or a rosin amine and a fatty acid of 10-20 C or a rosin acid. This removes the tendency of the pigment to agglomerate. C.O.C.

#### α-Ferric Oxide (C.I. 77491)

Columbian Carbon Co. USP 2,935,379 (13 June 1957)

α-Ferric oxide is obtained without need of seed crystal or calcination by digesting γ-ferric oxide at 70-100°C in an aqueous solution of 0.1-0.5 mole/l. of a ferrous salt. The resulting red pigment has excellent texture, dispersibility, tinting strength, and uniformity of colour. C.O.C.

#### Wet-milling Rutile Titanium Dioxide (C.I. Pigment White 6)

Natural Lead Co. USP 2,933,408 (28 May 1957)

Before the pigment is placed in the mill it is leached with water to reduce the occluded water-soluble metal salts to < 0.25% (as SO<sub>3</sub> on TiO<sub>2</sub>). The leached pigment is then readily made into an aqueous slurry in presence of 0.1-0.6% of a dispersing agent on weight of the pigment. C.O.C.

#### Converting Titanium Dioxide (C.I. 77891) from the Anatase to the Rutile Form

LT BP 865,327 (3 Dec 1958)

The anatase is calcined at 870-960°C with 0.1-5.0% of its weight of BPO<sub>3</sub> until < 90% is converted into the rutile form. C.O.C.

#### Yellow Ceramic Pigments

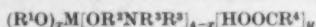
National Lead Co. BP 862,605 (25 Sept 1959)

Incorporation of a small amount of B<sub>2</sub>O<sub>3</sub> when making a ZrO<sub>2</sub>-V<sub>2</sub>O<sub>5</sub> pigment yields a very bright yellow glaze colour even in presence of SiO<sub>2</sub>. C.O.C.

#### Dispersing Agents for Pigments

Union Carbide Corpn. BP 863,412 (U.S.A. 17 Apr 1956)

Addition of a compound of formula—



(R<sup>1</sup> = Alk of 1-18 C or alkaryl, aralkyl or Ar of 6-10 C; R<sup>2</sup> = alkylene; R<sup>3</sup> = H, Alk or hydroxalkyl of 1-4 C or alkaryl, aralkyl or Ar of 6-10 C, the two R<sup>3</sup> may be the same or different; R<sup>4</sup> = Alk or alkenyl of 10-18 C; M = Si, Sn or Pb; x = 0, 1, 2 or 3; y > 4 - x, e.g. dibutoxydi(ethanolaminoethoxy) - silane - NN'-dioleate, renders pigments, e.g. TiO<sub>2</sub> (C.I. Pigment White 6), readily dispersible in paint and ink vehicles. C.O.C.

#### Interactions of Surface-active Agents with Congo Red (III p. 257)

#### Some Dyes Solubilised by Thiolsulphate Groups (VIII p. 267)

#### Fluorescent Brightening of Cellulosic Materials (VIII p. 268)

#### Yellow Dye Images by Colour Development (IX p. 269)

#### Fluorescent Brightening of Paper (XI p. 271)

#### Fluorescent Brightening Agents and their Behaviour in the Paper Industry (XI p. 271)

#### Characterisation of Sulphur Dyes (XIV p. 274)

## V—PAINTS; ENAMELS; INKS

### Lacquer Coating of Aluminium-Magnesium Alloys

R. D. Guminski and F. M. P. Meredith

J. Oil & Col. Chem. Assoc., 44 (Feb 1961) 93-110

Failure of lacquer coatings occurs more readily on Al-Mg alloys than on other Al alloys. The main cause is the presence of a hard, brittle, and slightly alkaline oxide film formed on the sheet during normal production processes. The greater the Mg content of the alloy the more prone is the lacquer to failure. Production techniques cannot be altered to yield sheet with < 0.5% Mg that is sufficiently oxide-free to be satisfactorily coated with ordinary commercial lacquers. Special lacquers can be compounded which do not fail when applied to alloys containing > 2% Mg; they are not, however, readily available, and the choice of materials is restricted. Poor lacquer adhesion can be overcome by acid, chemical, or electrochemical pretreatment, and the adhesion of commercial lacquers to Al-Mg alloys so pretreated is at least as good as that to any other Al alloy. Anodic pretreatment in H<sub>2</sub>SO<sub>4</sub> is particularly efficient, and most generally applicable, but chemical pretreatment with HNO<sub>3</sub> may be preferred for a small production line where the cost of an electrolytic plant is not justifiable. J.W.D.

### Coconut Oil-Isophthalic Alkyls for Stoving Enamels

J. Beikmanis, L. Kovacs, and R. H. Russell

J. Oil & Col. Chem. Assoc., 44 (Feb 1961) 150-155

The formulating and processing of short coconut oil-isophthalic acid alkyls as a function of polyol type and excess are discussed. The isophthalic-type alkyls are comparable with normal phthalic-type resins but are superior only in relation to mar-resistance, in which they are slightly better. Their limited compatibility with amino resins is a serious drawback. J.W.D.

### Water-dilutable Stoving Finishes

A. G. North

J. Oil & Col. Chem. Assoc., 44 (Feb 1961) 119-149

The use of stoving finishes that are partly or wholly thinned with water has both technical and economic advantages, but some problems arise; these are discussed in detail. Adequate degreasing of the metal surface and sufficiently long stoving at a temp. high enough to remove the water are essential. The relative merits of emulsion and solution systems are discussed, and the practical formulation and use of water-based coatings are illustrated by reference to new types of water-soluble alkyd-phenolic and alkyd-amino resin combinations. The performance of these new finishes is generally similar to that of conventional stoving finishes. J.W.D.

### Behaviour of Titanium Dioxide Pigments in High-speed Impeller Dispersion Mills

D. G. Dowling

J. Oil & Col. Chem. Assoc., 44 (Mar 1961) 188-208

The optimum milling conditions have been determined for dispersing a typical refined titania (Tioxide R-HD) in a typical long oil alkyd medium (Paralac 30W) in a laboratory mill (the Cowles Dissolver, Model 1-VG). By making use of the optimum values of the formulation variables (vol. concn. of pigment in mill-base, and the mill-base resin solids concn.) and the geometric variables (container diameter, charge depth, and impeller location) the time of milling can be minimised and, in addition, lower mill speeds may be used with possible savings in power. Limited study of other systems indicates that the same principles of formulation are valid, though the medium may have a marked effect on the quality of pigment dispersion. J.W.D.

### Irradiation Resistance of Varnishes

F. B. Waddington

J. Oil & Col. Chem. Assoc., 44 (Mar 1961) 179-187

The effect of irradiation on varnishes and related products is discussed; the experimental techniques employed are described. Varnishes fall into three categories: (a) those based on radiation-sensitive materials such as cellulose acetate or nitrate, which will not withstand much more than 100 Mrad; (b) most other varnish bases such as alkyds, drying oils, phenolics, and polyesters, with a substantial resistance to irradiation but for which a "useful life" dose should be determined by experiments closely related to actual service conditions; (c) a few radiation-resistant varnishes, such as those based on

diphenyl siloxane, or epoxy resins cured with an aromatic catalyst, which are stable to dosages of up to several thousand Mrad over a range of conditions. High radiation resistance appears to be associated with the presence of a large proportion of aromatic rings in the molecule; conjugated double bonds and ionic linkages also have a stabilizing effect.

J.W.D.

#### Stability of Latex Paints in Different Climates

J. A. Gordon Jr.

*Deutsche Farben-Zeitschrift*, 15 (Jan 1961) 11-13

Properties and compositions of the principal types of latex paint are summarised, earlier experiments on suitable systems are described, and Lytron 680 is used as an example. Pigment dispersion materials, suitable emulsions for additions, and proposals for pigment combinations for internal and external painting are given.

*Ibid.*, 15 (Feb 1961) 67-71

An exhaustive review of the properties of Lytron 680 is given.

H.H.H.

#### Underrusting of Painted Steel—Occurrence, Assessment, and Treatment

J. A. W. van Laar

*Deutsche Farben-Zeitschrift*, 15 (Feb 1961) 56-67

Under practical conditions, and in lab. tests, cold-rolled steel when painted properly does not show rusting through, but underrusting takes place at uncovered and damaged spots and clipping edges. In the motor-car industry, about 31 paint systems have been developed to combat such development, and tests have revealed distinct differences in speed of underrust. The causes are found to result from several elementary processes which are analysed and discussed. Rusting of painted steel constructions is dependent on passivation as well as on thickness of the paint layer.

H.H.H.

#### Corrosion Protection by Pigments

J. D'Ans and V. Grooppe

*Deutsche Farben-Zeitschrift*, 15 (Feb 1961) 51-55

The Pb, Ba, Zn, Cd, and Ca chromate pigments used as protection against corrosion are reviewed. 13 References.

*Ibid.*, 15 (Mar 1961) 99-104

The following conclusions are now drawn: (1) The pigment should preferably have a basic reaction; (2) Every suitable pigment which undergoes alteration in a paint, whether by chemical or electrochemical reactions, to produce an insol. cpd. can form protective layers; (3) Oxidising pigments must have such an oxidation potential as not to oxidise unduly the organic binding medium; (4) The efficiency of the protective pigment depends on its solubility; (5) Decisive qualities of a cover layer are electrical resistance, adhesive power, indifference to impurities, and slow ageing. Addition of slightly sol. phosphates can improve the protective action of chromate cover layers; (6) Binding agents have the function of protection against damage; (7) Electrochemical problems arise after the formation of the cover layers; (8) The known layers are all basic oxides, oxyhydrates, or basic salts, which form extremely thin three-dimensional networks whose stability depends on supporting cpd. Warning is expressed against the injurious effects of water-sol. electrolytes if present in the pigment.

H.H.H.

#### Inhibitors, Softeners, and Additives in Artificial Resin Lacquers

B. Waesser

*Deutsche Farben-Zeitschrift*, 15 (Jan 1961) 13-17

This subject is reviewed and 34 references are cited. It is pointed out that most auxiliaries used for steel paints have little effect on corrosion unless they contain ester groups or are in a pre-oxidised condition, when they facilitate adhesive and covering power and diminish tendency to corrode. Resinous lacquers for steel painting should be in a pre-oxidised state.

H.H.H.

#### PATENTS

##### Cellulosic Coating Compositions

DuP

BP 865,267 (U.S.A. 10 Mar 1958)

Coatings having very high resistance to organic solvents and corrosive chemicals are yielded by a composition comprising an organic-solvent solution of a cellulose derivative, a compound containing at least one 1,2-epoxide group, and a urea- or triazine-formaldehyde resin as curing agent for the epoxide compounds.

C.O.C.

##### Sprayable Pigmented Polyvinyl Acetal Composition

DuP

BP 865,114 (U.S.A. 21 June 1956)

A composition suitable for spraying on to transparent materials to tint them consists of a pigment, a polyvinyl acetal resin of average mol. wt. < 300,000 and a solvent. The pigment must have a refractive index < 2.3 at the wavelength of light giving maximum light transmittance through the pigment, and 95% by weight must have particle size < 1  $\mu$ . The pigment must form < 15% by weight of the composition.

C.O.C.

##### Coating Compositions based on Methyl Methacrylate Polymers, Copper Bronze (C.I. Pigment Metal 2) and a Transparent Pigment

DuP

USP 2,934,512 (20 Sept 1954)

A composition which does not gel during storage and which yields coatings of improved resistance to crazing comprises a methyl methacrylate polymer or copolymer, a solvent, a transparent coloured pigment and 0.5-35.0% of copper bronze flake. The composition yields polychromatic coatings in which the transparent pigment provides the basic hue and the bronze flakes provide coloured mirrors that reflect various elements of the basic hue.

C.O.C.

##### Organosilicon Resin Coating Compositions

Midland Silicones

BP 862,470 (U.S.A. 30 June 1958)

A composition which neither gels nor skins on standing in a closed container but which cures in < 24 h at room temperature is made up of (1) a hydroxyl-free polysiloxane having 1.1-1.7 monovalent hydrocarbon radicals per Si atom and containing Si-bonded acyloxy radicals of < 4 C, there being 1 acyloxy radical per 3-100 Si atoms, and (2) a monohydrocarbortriacyloxysilane in which each hydrocarbon radical is of < 4 C.

C.O.C.

##### Organopolysiloxane Varnishes

Société des Usines Chimiques Rhône-Poulenc

BP 862,602 (France 5 Aug 1958)

Coatings which give excellent protection to painted surfaces and which are readily cleansed with wetting agents or caustic alkali comprise (a) 30-60% of a partially condensed organopolysiloxane having 1.2-1.6 hydrocarbon groups per Si atom and 0.5-2.5 Si-bonded hydroxyl groups, (b) 1-25% of a methyl or ethyl orthosilicate, (c) 1-20% of an organic-solvent-soluble titanate or zirconate ester, (d) 0-20% of an organic-solvent-soluble organic zinc salt, and (e) < 15% of a solvent for all the other components.

C.O.C.

##### Pelleting Lacquer (I p. 257)

Manufacture of Aluminium Hydroxide (C.I. 77002) as a

Pigment for Printing Ink (IV p. 259)

Dispersing Agents for Pigments (IV p. 262)

Methods of Testing Certain Mechanical Properties of Lacquer Films (XIV p. 274)

## VI—FIBRES; YARNS; FABRICS

### Cyanoethylation of Cotton in Aqueous Medium

N. M. Bikales and L. Rapoport

*Text. Rec.*, 78 (Mar 1961) 71-74

Cotton cyanoethylates in aqueous medium much slower than in conventional processes, and the maximum degree of cyanoethylation obtainable is far short of full cyanoethylation. Under optimum conditions (15-20°C) using 10-12% NaOH, at a liquor ratio of > 10 and in presence of an additive, e.g. NaCl, the amount of acrylonitrile lost in side reaction about equals that which reacts with the cotton. Cotton cyanoethylated in this manner in absence of tension, unlike ordinary cyanoethylated cotton, lacks resistance to microbiological attack. Probable factors in causing this are deorientation of the cellulose and partial hydrolysis of cyanoethyl groups.

C.O.C.

### Response of Cottons to Chemical Treatments. I—Fibre Mercerisation and Urea Treatments

L. Rebenfeld

*Text. Research J.*, 31 (Feb 1961) 123-130

Many changes in fibre properties on chemical treatment can be interpreted in terms of fibrillar orientation of the fibres as estimated by the X-ray angle. Under mercerising conditions cottons with high X-ray angles underwent greater extension in fibre length and elastic modulus and greater decrease in fibre breaking extension than did cottons with low X-ray angles. Changes in fibre breaking tenacity could not be related to known structural features of the cotton fibre.

C.O.C.

## Sunlight Degradation of Various Types of Rayon Staple

R. Kotake and T. Okamoto

### I—Sunlight Degradation of Rayon Staple

*J. Soc. Textile Cellulose Ind. Japan*, 17 (Jan 1961) 11–16

Degradation of various types of rayon staple on exposure to sunlight and Fade-Ometer has been studied, and the following conclusions are drawn—(a) All types of rayon staple examined showed similar degradation curves on exposure to sunlight. Of these, high-tenacity rayon staple had the highest strength and elongation after 400 h exposure. (b) From strength and elongation data on irradiated crimped delustrated rayon, correlation between the time of Fade-Ometer exposure and amount of sunlight irradiation has been established. This relation is expressed by the following empirical equations—

- (i)  $y = 21.7x$  (from strength data)
- (ii)  $y = 16.7x$  (from elongation data)
- $y$  = time of Fade-Ometer exposure (h)
- $x$  = amount of sunlight irradiation (kcal/cm).

### II—Sunlight Degradation of Rayon Staple Fabrics

*Ibid.*, 16–20

Fabrics (standard muslin—No. 9 standard structure) woven from high-tenacity rayon staple, regular rayon staple, and cotton were exposed to sunlight for 5 months under identical conditions. From a study of the change in dry and wet tenacity, dry and wet elongation, tear strength, and flexing abrasion of the irradiated samples, high-tenacity rayon staple is shown to be superior from a practical standpoint to cotton and regular rayon staple. L.G.L.

### Effect of Soaking Time on Recovery Properties of Yarns and a Fabric of Secondary Cellulose Acetate Immersed in Water at 20°C

J. C. Guthrie and (Mrs.) S. Norman

*J. Textile Inst.*, 52 (Feb 1961) T96–T99

The yarn elastic recovery and the fabric crease recovery depend on the duration of soaking before measurements are made. Both increase with increasing soaking time, but values are comparatively low, even after long soaking times. The measured wet modulus is very sensitive to rate of extension or rate of loading. W.R.M.

### Fission of Triphenylmethyl Derivatives of Secondary Cellulose Acetates by Hydrogen Bromide

W. R. D. Leigh

*J.C.S.*, (Feb 1961) 754–756

Previous attempts by Malm *et al.* (*J. Amer. Chem. Soc.*, 70 (1948) 2740) to prepare secondary cellulose acetates with all the free OH groups in the primary 6-positions via the 6-triphenylmethyl deriv. failed owing to acetyl migration detritylation. It is now established that, with HBr in anhyd. acetic acid as the detritylating agent, this migration is suppressed and secondary cellulose acetates are made available with up to 90% of the free OH groups in the primary positions. This use of HBr extends the range of free-hydroxyl distributions and affords products with interesting solubility characteristics. H.H.H.

### $\alpha \rightarrow \beta$ Transformation in Keratin

A. Skerthly and H. J. Woods

*J. Textile Inst.*, 51 (Dec 1960) T517–T527

The equatorial scatter of X-rays by Lincoln wool fibres stretched by up to 60% in water at room temperature has been recorded. There is an important change in scattering at extensions of less than 20% and this is ascribed to a single-phase transformation from crystalline  $\alpha$ -keratin to crystalline  $\beta$ -keratin. Except in unstretched and fully stretched conditions, there is a deficit in the total amount of crystalline keratin, indicating the existence of an intermediate state in which the keratin is not sufficiently well-ordered to contribute to the 10 or 4–65 Å equatorial peaks. The difficulty of transforming the Pauling-Corey AB<sub>2</sub> cable is recalled, and the possibility of packing coiled-coil  $\alpha$ -helices without their forming twisted cables is pointed out. M.T.

### Crystallographic Changes in Wool Keratin during Chemical Modification, especially Finishing

A. Skerthly *J. Textile Inst.*, 51 (Dec 1960) T528–T543

A new rapid-scanning X-ray diffractometer is described and its practical and theoretical limitations are discussed. Its use in a preliminary investigation of the kinetics of lattice degradation in wool keratin is reported. Wool fibres were treated with (a) 98% formic acid, (b) 10-M lithium

bromide, (c) 30% phenol, (d) setting agents (urea-reducer). It is concluded that setting treatments do not appreciably affect the stability of the crystalline keratin lattice, but increase the degree of crystalline perfection in the lattice, i.e. they improve the stability of the "amorphous" components in the system. Estimates are given of the free energy of activation for perturbation of the keratin crystal lattice for native and modified wool, and the probable reaction mechanisms of the reagents used are briefly discussed. M.T.

### Ultraviolet Absorption Spectrum of Solid Keratin

E. G. Bendit *J. Textile Inst.*, 51 (Dec 1960) T544–T561

The u.v. absorption spectrum has been determined of native keratin in the form of thin, longitudinal sections of horsehair. The absorption peak at pH 7 is at 278–279 m $\mu$ , compared with 274.5 m $\mu$  for the principal absorbing amino acid, tyrosine, i.e. there is interaction between the tyrosyl OH and a neighbouring group. The position of the peak is unaffected by treatment at 20°C with acid at pH 1.5 or with unbuffered 8-M urea, but is shifted to approx. 275 m $\mu$  by 8-M LiBr. Under the conditions used, only LiBr produces supercontraction. Treatment with hot NaOH or exposure to u.v. radiation increases absorption, particularly above 300 m $\mu$ . The variations observed in position of the tyrosine peak indicate that caution must be used in making inferences about the environment of the tyrosine residue in the native protein from the spectrum of keratin in solution. M.T.

### Sulphur Compounds in Acid Hydrolysates of Wool

Barbara Lewis, A. Robson, and Eva M. Tiler

*J. Textile Inst.*, 51 (Dec 1960) T653–T664

An investigation was carried out to determine the chemical properties, origins, and approximate amounts of the sulphur-containing compounds in acid hydrolysates of wool. It is concluded that (a) methionine and cystine are probably the only two sulphur-containing amino acids in wool, although cysteine may be present, (b) cystine in wool cannot be accurately determined as cysteic acid after oxidising wool with peracetic acid, (c) there are five sulphur-containing compounds in acid hydrolysates of wool, including cysteine, that appear to be derived from cystine during hydrolysis, and (d) until all the sulphur-containing compounds in wool hydrolysates have been identified, the possibility remains that cystine and methionine are not the only sulphur compounds present. Because of (c), the presence of cystine in wool hydrolysates cannot be regarded as evidence for its presence in the wool protein structure. M.T.

### Study of Problems in Protein Chemistry by Non-hydrolytic Methods for Determination of Thiol and Disulphide

J. A. MacLaren, S. J. Leach, and J. M. Swan

*J. Textile Inst.*, 51 (Dec 1960) T665–T677

Present methods for estimating SS and SH groups in insoluble proteins are unsatisfactory. Methods have been devised whereby SH groups are estimated by treating the protein with MeHgI, and (SS+SH) by treating with MeHgI or HgCl<sub>2</sub> in presence of sulphite. These analytical techniques have been applied to analysis of keratins, irradiated wool, and oxidised wool. Relatively large amounts of partially oxidised disulphide residues have been detected in oxidised wool. The principles of these new methods have been applied to the study of disulphide-splitting reactions, to preparation of wools from which SH and SS groups have been removed, and to labelling of proteins at the SH and SS residues. M.T.

### Relation between Permanent Set and Covalent Cross-linking in Alkali-treated Wool

P. T. Speakman

*Trans. Faraday Soc.*, 57 (Mar 1961) 518–523

Permanent set and supercontraction of keratin fibres in alkali are directly proportional to the square root of the loss of cystine in the alkali treatment. Cystine is converted to covalent lanthionine links. A similar proportionality between the isotropic, amorphous polymer length and the square root of the degree of cross-linking has been predicted for randomly cross-linked axially oriented polymers and successfully applied to rubber and polyethylene. The differences between cross-linked fibrous proteins and cross-linked rubber and polyethylene, and their theoretical implications are discussed. W.R.M.

**Fleece-rot Discoloration in Greasy Wool**J. R. Paynter *J. Textile Inst.*, 52 (Feb 1961) T64-T72

The incidence of fleece rot and its association with some wool characters have been investigated, using results from five flocks of Merino and Merino Corriedale crossbred sheep. Greasy wool colour showed a highly significant correlation with fleece rot. Quality number and staple length showed a small significant correlation, but wool wax and suint were not quantitatively related to wool colour and did not reflect the relationship of wool colour to fleece rot.

W.R.M.

**Supercontraction of Wool in Aqueous LiBr; Inhibition by Free Br<sub>2</sub> and its Relation to Thiol-Disulphide Interchange**

W. G. Crewther and L. M. Dowling

*Text. Research J.*, 31 (Jan 1961) 31-37

Wool whose thiol content has been decreased by 70% by treatment with *N*-ethylmaleimide supercontracts much more slowly than untreated wool at 98.5°C in 6-M LiBr at pH 6, whereas in 4-M LiBr/1-N HCl the rates of contraction of treated and untreated wool are the same. Pretreatment with dil. aq. bisulphite accelerates supercontraction much more in 4-M LiBr at pH 8 than in 4-M LiBr/1-N HCl. Apparently, interchange reactions between thiol and disulphide groups facilitate supercontraction. Presence of Br<sub>2</sub> in the LiBr solution used causes different effects depending upon pH. In 1-N HCl supercontraction is accelerated at all levels of contraction; at pH 4-5 initial supercontraction is accelerated but the second stage retarded; at pH > 6 supercontraction is retarded at all levels of contraction. Wool supercontracts in dil. HCl solutions of Br<sub>2</sub> even at low temperatures. Br<sub>2</sub> reacts with the thiol groups during supercontraction in LiBr solutions and acts at a site in the fibres affected by the *N*-ethylmaleimide treatment. Wool reduced with thioglycolate and either cross-linked with ethylene dibromide or treated with CH<sub>3</sub>I shows no change in contraction kinetics when Br<sub>2</sub> is added to the LiBr solution. Seemingly Br<sub>2</sub> inhibits supercontraction in LiBr solution by reacting with thiol groups and so interfering with interchange reactions between the thiol and the disulphide groups. C.O.C.

**Supercontraction and Solubility of Alkali-treated Wool**K. J. Whiteley *Text. Research J.*, 31 (Jan 1961) 82-83

Treatment of wool with dilute alkali, e.g. 0.05-M K<sub>2</sub>CO<sub>3</sub> at 50°C for 1 h, produces significant alterations in the fine structure, resulting in decreased urea-bisulphite and peracetic-ammonia solubilities and in supercontraction.

C.O.C.

**Solubility of Wool in Bromine Water**

M. Laveau and M. Caillat

*Bull. Inst. Text. France*, (91) (Nov-Dec 1960) 101-108

From a study of the effect of bromine concentration, pH, temp. and time of treatment on the solubility of wool in bromine water, the authors adopt the following standard conditions—0.5 g wool in 50 ml 2% bromine water, 17°C for 1 h, followed by filtration and washing with NaHSO<sub>3</sub> soln. and water. Solubility of untreated wool is 22%. The effect of different treatments on the "bromine solubility" is, in general, not sufficiently great to make the test of wide applicability. However, fairly severe treatments with free halogens or alkali hypohalites lead to characteristically reduced "bromine solubility". J.C.F.

**Physical Properties of Wool Fibres at Various Regains**

A. R. Haly and M. Feughelman

**I—Stresses developed at Constant Strain owing to Regain Change by Absorption or Desorption***Text. Research J.*, 31 (Feb 1961) 131-134

Fibres held at constant extension of 1.5 or 20% and subjected to various changes in r.h. develop very different stresses according as equilibrium at a given r.h. is approached by absorption or by desorption of water by the fibre. At 1.5% extension, when approach was by absorption, for all r.h. used the stress fell to below that when the fibre was in water and then increased. A qualitative explanation is given in terms of changes analogous to sol-gel transformations.

**II—Recovery from Extension***Ibid.*, 135-140

Under all conditions of extension and r.h. used, length recovery occurred in two stages. Early, rapid recovery was

followed by a slow recovery which was, in most circumstances, linear with log (time). The amount of rapid recovery depends on the stress in the fibre at the instant of release. Recovery from 20% extension is greatly accelerated as r.h. rises above 60%, probably because of sharp increase in the amount of "free" water in the fibre. Recovery from 1.5% extension is different in character, perhaps because recovery from low extension may contain a large elastic component. C.O.C.

**A Fatty Acid-Protein Complex in Human Hair**

A. W. Holmes

*Nature*, 189 (18 Mar 1961) 923

A fatty acid-protein complex has been isolated from ethanol extracts of human hair. This complex, which is insoluble in non-polar solvents, maintains the hair intact when subjected to papain-bisulphite digestion. C.O.C.

**Studies on Rabbit Fibres. XXXIX—Relation between Carrotting Conditions and Various Physical Properties of Fibres in the Carrotted Bunks**

S. Ikeda

*J. Soc. Textile Cellulose Ind. Japan*, 17 (Jan 1961) 21-26

In order to test statistically the effects of carrotting conditions on physical properties, random samples of test fibres were taken out of the carrotted bunks. Changes in mechanical and frictional properties and in crimps were detected. The method of sampling and estimation employed will be of use in the analysis of the relation between the feltability and the physical properties of the bulks. I.G.L.

**Variation of Length of Moistened Polyamide Fibres with Time**

G. Kunzmann

*Faserforsch. und Textiltech.*, 12 (Feb 1961) 61-66

The variation in length following sudden moistening of polyamide fibres has been measured using a mechanico-optical apparatus by means of which a voltage proportional to change in length can be automatically recorded as a function of time. Fibres of differing thickness were examined over the temperature range 20-80°C. The beginning of the change in length does not coincide with that of water absorption and this retardation is theoretically considered as a relaxation process. W.R.M.

**New Ternary Solvents for Polyamides**

V. Gröbe

*Faserforsch. und Textiltech.*, 12 (Feb 1961) 78-79

Mixtures of aqueous solutions of zinc chloride, hydrochloric acid, and acrylonitrile or propionitrile or acetonitrile dissolve unstretched polycaprolactam fibres at room temperature. W.R.M.

**Modification of Crystalline Structures of Nylons 6.6 and 6 by Iodine**

T. C. Tranter and R. C. Collins

*J. Textile Inst.*, 52 (Feb 1961) T88-T91

Treatment of nylons 6.6 and 6 with aqueous solutions of potassium iodide containing iodine results in a marked change of equatorial reflections and the appearance of new meridional reflections in the X-ray diffraction patterns. It is suggested that these changes result from an ordered association of iodine with polymer molecules and that the amide group is most likely to form a complex with iodine or the tri-iodide ion. W.R.M.

## PATENTS

**Flame-resistant Cellulose Ester Fibres**

Eastman Kodak Co.

USP 2,933,402 (12 Dec 1960)

Cellulose esters of organic acids are made non-inflammable by incorporating an amphoteric metal salt of bis( $\beta$ -chloroethyl) phosphate. Thus cellulose acetate fibres containing 1% by weight of Al, Ti, or stannous phosphate do not burn when exposed to flame. C.O.C.

**Cellulose Acetate having Antiseptic Properties**

Sangyokuse Kabushikikaisha

BP 862,515 (Japan 16 Dec 1957)

Addition to the spinning solution of an organotin compound (0.01-0.20% on wt. of cellulose acetate), e.g. tribenzylphenyl tin, results in fibres which inhibit the growth of microbes. C.O.C.

**Improving Light-stability of Polyamides**

Vereinigte Glanzstoff-Fabriken

BP 862,577 (Germany 25 Apr 1958)

Incorporation of the complex obtained by reaction of

a salt of Co, Ni, Cr, Cu, or Mn with an alkali-metal polyphosphate much improves the stability to light of polyamide filaments. It gives excellent protection to polyamides delustrated with  $\text{TiO}_2$  without affecting their whiteness or mechanical properties. C.O.C.

#### Irradiation of Nylon

General Electric Co. BP 860,280 (U.S.A. 21 Nov 1956)  
Irradiation of nylon with high-energy ionising radiation having energy equivalent to  $< 5 \times 10^4$  eV to a radiation dose of  $1 \times 10^5$  to  $1 \times 10^6$  rep. at  $60^\circ\text{C}$  to just below m.p., confers reduced susceptibility to oxidation. In a modified process the nylon is irradiated at  $< 100^\circ\text{C}$  and is then heated to just below m.p. Cross-linked nylon produced by these methods has permanently reduced elongation but retains its original tensile strength. C.O.C.

#### Heat-resistant Polyvinyl Alcohol Fibres

FH BP 864,308 (Germany 16 Jan 1957)  
Addition to an aqueous spinning solution of polyvinyl alcohol of 0.3–10.0% of an alkali-metal phosphate of acid reaction and/or a condensed alkali-metal phosphate of acid, neutral, or alkaline reaction, and then spinning by either wet or dry process, yields filaments which are fast to boiling and resistant to heat. C.O.C.

Alginate Acid and its Behaviour towards Cold-dyeing Reactive Dyes of the Dichlorotriazine type (VIII this page)

Setting of Wool Fibres in Dyeing (VIII p. 267)

Fluorescent Brightening of Cellulosic Materials (VIII p. 268)

Pigmented Viscose Rayon (VIII p. 268)

Improving the Stability to Heat of Polyacrylonitrile Fibres (X p. 270)

Vinyl Polymers containing a Chemically Condensed Azoic Coupling Component (XIII p. 273)

Infrared Spectra of Polypeptides in Various Conformations: Amides I and II Bands (XIV p. 274)

Sulphur-containing Compounds—Nomenclature (XV p. 276)

## VII—DESIZING; SCOURING; CARBONISING; BLEACHING

### Desizing with Sodium Bromite

R. Freytag *Melliand Textilber.*, 42 (Feb 1961) 193–198  
Laboratory trials and large-scale experiments showed that cotton goods containing starch can be desized with sodium bromite ( $\text{NaBrO}_2$ ) and that removal of starch is as complete as by enzymes. Treatment was carried out at room temp. and pH 10. Bromite consumption was about 350 g  $\text{NaBrO}_2$  (100%) per metric ton of cotton goods. Comparison of the DP of goods desized with bromite and enzymes respectively showed no difference. Stainless (V4A) steel after having been immersed for 1000 h in a boiling solution containing 10 g/l. active bromine did not seem to be corroded. W.M.

### Stabilisation of Hydrogen Peroxide Bleaching Baths for Bleaching of Wool

H. Baier *S.V.F. Fachorgan*, 16 (Jan 1961) 72–79  
The stabilising effect of ammonia, sodium pyrophosphate, disodium hydrogen phosphate, trisodium phosphate, and Calgon T on 1% hydrogen peroxide solutions in the pH range 9.1 to 9.7 at  $45^\circ\text{C}$  is investigated. Various types of water, i.e. soft, hard, iron-containing, etc., are employed to show the effectiveness of the above stabilising agents in these conditions. P.B.S.

### Colour Effects Produced in Nearly White Fabrics by Household Detergents

M. S. Furry, P. L. Bensing, and M. L. Johnson *Amer. Dyestuff Rep.*, 50 (23 Jan 1961) 50–55  
Fluorescent and non-fluorescent colour effects produced in nearly white unsoiled fabrics of cotton, Dacron, and nylon by repeated laundering with household detergents containing pigments and fluorescent brightening agents were evaluated by the colour difference meter, modified with filters. Measurements effectively indicated the colour changes and the contribution of fluorescence to whiteness, and these results compared well with visual observations made for whiteness under north-sky daylight or the daylight lamp with added ultraviolet radiation and for brightness under ultraviolet radiation. Presence of pigments

caused greying on cotton, the extent depending on the finish present, but nylon and Dacron were unaffected. Fluorescent brightening agents markedly whitened unfinished cottons but their effectiveness on finished cotton was variable, on nylon the effect was to mask some of the yellowness, and on Dacron they had little effect. S.B.D.

Simultaneously Dry Cleaning and Imparting Water-repellency to Garments (X p. 270)

## VIII—DYEING

### Alginate Acid and its Behaviour towards Cold-dyeing Reactive Dyes of the Dichlorotriazine type

M. Ulrich *Melliand Textilber.*, 42 (Jan 1961) 81–85 (Feb 1961) 205–209

Alginate acid is stronger than acetic, but can be pptd. by monochloroacetic acid. Alginate acid can be etherified as well as esterified. Ca alginate fibre has—if absolutely dry—a higher tensile strength than ordinary viscose rayon, but the wet strength is very low. The moisture content and the sp. gr. increase with increasing Ca content. Swelling in water increases diameter by  $\sim 50\%$ ; in 2% NaOH by  $\sim 100\%$ . Calgon swells and dissolves the fibre very quickly. If fibre is mounted in water and Calgon is slowly added, differences in speed of dissolution indicate variation in density of different fibre layers. If treated with conc.  $\text{H}_2\text{SO}_4$  or  $\text{NH}_4$ -oxalate the fibre dissolves and arrangement of Ca cryst. allow conclusions to be drawn regarding fibre structure. Ca alginates react with cold-dyeing reactive dyes even in the absence of alkali, since alkali is formed by slow hydrolysis of the fibre. Alginate acid contains only secondary alcohol groups and, since it is irreversibly dyed by Procion dyes, secondary alcohol groups (and not only primary) must be able to react with these dyes. Benzoylated Ca alginate gives—in contrast to the untreated fibre—only pale shades with Procion Blue 3GS, indicating again that covalent linking takes place between dye and secondary OH groups. W.M.

### Dyeing Methods for Remazol Dyes

E. Ungermann *Melliand Textilber.*, 42 (Jan 1961) 89–96  
These dyes can be dyed by exhaustion processes with addition of salt and alkali. Several padding methods can be used. They may be divided into two groups: those with and those without a drying operation. The most important processes belonging to the first group are (a) the two-bath pad-steam and (b) the one-bath pad-dry process. For intermediate drying hot flue or nozzle dryers are recommended. Stenters are less suitable (marks from clips or pins). There is a risk of dye migrating during drying, and addition of thickeners is useful. In the two-bath pad-steam process the fabrics are padded with dye soln., dried, padded with salt + NaOH soln. and steamed at  $103\text{--}105^\circ\text{C}$  for 20–30 s. In the one-bath process the goods are padded with a cold solution of dye, urea, and soda, and then dried, whereby fixation of dye takes place. Time of drying depends on temp.—at  $100^\circ\text{C}$ , 5–8 min, at  $140^\circ\text{C}$ , 1–2½ min. Processes without intermediate drying include the one-bath pad-steam process. The goods are padded with a solution containing dye, equal amounts of soda and bicarbonate. The goods are then steamed at  $103\text{--}105^\circ\text{C}$  for 80–100 s. The padding soln. can be kept for 3–4 h. In the one-bath pad-batch process fixation of the dye takes place at room temp. The strongly alkaline soln. ( $\text{NaOH} + \text{Na}_2\text{PO}_4$ ) is of limited stability and can be kept—depending on the dye—for 20–120 min. After being padded the goods are batched up and are left for 4–24 h. Rotation is not necessary. In another version, soln. of dye and alkali are prepared separately and are mixed only just before being fed into the small padding box. The problem of stability of the bath does not arise here. Instead of the pad-batch process the pad-roll process (Rydholm system) can be used. W.M.

### Dyeing of Vat Dyes by the Pigment-padding Process and Factors Determining Levelness

R. Loes *S.V.F. Fachorgan*, 16 (Jan 1961) 59–63  
A theoretical analysis of the process. Levelness is shown to depend mainly on grain size of the dispersion, wetting of the fabric, immersion time, quantity of padding liquor in the developing bath, and developing time. P.B.S.

### Studies in Adsorption. XIV—Mechanism of Adsorption of Disperse Dyes by Cellulose Acetates and other Hydrophobic Fibres

C. H. Giles *Text. Research J.*, 31 (Feb 1961) 141–151

Consideration of recent quantitative data for adsorption of disperse dyes on cellulose acetates leads to the following conclusions. The dye probably enters the fibre partly in mono-disperse and partly in associated forms and penetrates between the fibre chains in regions inaccessible to water. This type of adsorption produces the characteristic linear adsorption isotherms. Several properties of the dye molecule determine the maximum adsorption of dye—(a) *Size* The smaller the molecule the more easily it can penetrate into the fibre structure, hence the higher the maximum adsorption. (b) *No. of H donor atoms* The more donor atoms there are the higher the adsorption in certain cases, the dye forming  $-OH \dots$  and  $-NH \dots$  bonds with the fibre. (c) *No. of H acceptor atoms* The dye forms  $-CH \dots O$ ,  $-CH \dots N$ , and  $-CH \dots Cl$  bonds with the  $CH_2$  groups of the acetyl groups in the fibre. Thus the  $-CH \dots Cl$  bond between ethyl acetate and chlorobenzene in aqueous ethanol has energy of about  $-3$  kcal/mole, and chlorobenzene itself is strongly adsorbed by cellulose triacetate from this solution. In some cases adsorption increases with the number of acceptor atoms, in others it decreases because of opposing electronic effects of other substituents. (d) *Steric effects and functionality* Wide spacing (e.g. 1,4-disubstitution) of bonding atoms tends to reduce adsorption, probably by promoting dye-dye association rather than dye-substrate association. Nylon and Terylene appear to adsorb by a similar mechanism, Terylene showing additional (aromatic nucleus) dye-fibre association. 51 References. C.O.C.

### Absorption of a Disperse Dye by Cellulose Acetate in Presence and Absence of Butanol

E. P. Teulings and H. J. White

*Text. Research J.*, 31 (Jan 1961) 52–56

Absorption of C.I. Disperse Red 1 by secondary cellulose acetate was studied when the dye was in true solution and its concentration remained constant. Aqueous alcohol baths with various additions of butanol were used at 50 or 65°C. Rate measurements were consistent with a diffusion process having a rate constant of about  $1 \times 10^{-11}$  cm<sup>2</sup>/s. The action of the butanol was complex. It caused a minimum in the equilibrium uptake of dye and in the rate of attainment of equilibrium as dye concentration in the bath increased. Possible reasons for the initial decrease are competition for absorption sites between dye and butanol or complex formation in the dyebath. Swelling with increased accessibility may account for the increase after the minimum. C.O.C.

### Some Dyes Solubilised by Thiolsulphate Groups

B. Milligan and J. M. Swan

*Text. Research J.*, 31 (Jan 1961) 18–25

Study of the affinity for wool of four thiolsulphate azo dyes (DSSO<sub>2</sub>Na) and three of the corresponding sulphonate dyes (DSO<sub>2</sub>Na) showed three of the thiolsulphate dyes to be unexpectedly good, especially as regards fastness to washing. Under certain conditions of application the thiolsulphate dyes partly decompose so that some dye becomes bound covalently to the wool and some is converted to the symmetrical dye disulphide (DSSD). C.O.C.

### Dyeing Wool Union Cloths in Acid Media

B. J. Pelle

*Tex.*, 19 (Nov 1960) 741–748

A one-bath process using a wool reserve agent, Trifol RZ extra, is especially recommended for plush and furnishing materials as giving extra protection to the wool fibre, excellent reproducibility, and the possibility of producing two-tone effects by a one-bath process, without affecting light-fastness. Clean union fabric is pretreated at 40°C for 10–15 min. with 10–20% sodium sulphate, 2–4% Trifol RZ extra (dissolved in warm water) and 6% acetic acid (pH 4.5–5.0). The dye soln. is then added and the temp. brought to near boiling in 45 min and maintained for 30–60 min. R.A.

### Setting of Wool Fibres in Dyeing

R. V. Peryman and R. F. Pickup

*J.S.D.C.*, 77 (Apr 1961) 149–154

### Oxidation Dyeing of Fur with Trihydroxynaphthalenes

D. I. Brozovskii and V. V. Kozlov

*Kozhevniko-Obuvnaya Prom.*, 2 (6) (1960) 18–20

*Chem. Abs.*, 55 (6 Feb 1961) 3065

White rabbit fur was dyed with 1,3,7-, 1,3,6-, and 1,3,5-trihydroxynaphthalene, individually and in mixtures, and with or without mordanting with Fe, Cu, or Cr. After being mordanted at 20°C the fur was dyed with 2 g dye/l. (1 g of each dye when mixtures were used) for 3 h at 33–37°C. These oxidation bases can be used alone but better colorations are obtained by mixing them with phenylenediamines or aminophenols. The dark grey and dark brown obtained with 1,3,7-trihydroxynaphthalene and *m*-aminophenol and *p*-aminophenol, respectively, on an iron mordant are of commercial importance. The dyeings have satisfactory fastness to light. These dyes were also successfully used on sheepskins. C.O.C.

### Monforts Reactor for Continuous Dyeing of Synthetic-fibre Fabrics

F. Fortess, J. E. Torbat, G. C. Ward, and J. S. Warner

*Amer. Dyestuff Rep.*, 50 (23 Jan 1961) P57–P62

The Monforts Reactor Machine (see *Amer. Dyestuff Rep.*, 49 (16 May 1960) 351) has great potential for economically and continuously handling short runs. In pilot trials major emphasis was placed on reserve, cross, and union dyeing of cellulose triacetate or secondary cellulose acetate with either cotton or rayon. Commercially feasible processes are available using selected direct dyes or reactive dyes on the cotton or rayon and disperse dyes for the cellulose acetate and triacetate using a common bath containing suitable swelling agents. Thus, for cellulose triacetate, the use of an aqueous bath containing 25% diacetone alcohol and 5% phenyl glycol ether makes possible heavy shades within 10 s. This same treatment raises the safe-ironing point from 190°C to 230°C, thus eliminating separate heat setting. A method has also been developed to obtain an azoic black. The use of the Monforts Reactor to process polyamide, polyester, and polyacrylonitrile fabrics is described and the advantages and disadvantages of processing various types of fabric are discussed. S.B.D.

### Disperse Dyes applied by the Thermosol Process

W. Beckmann and W. Wunder

*Melliand Textilber.*, 42 (Feb 1961) 198–205

Disperse and vat dyes are suitable for application to synthetic fibres by the Thermosol process. Basic dyes can be fixed on polyamide and polyacrylonitrile fibres, acid dyes on polyamide fibres. Fibre damage can be avoided. For mixtures containing wool or Perlon the temperature should be kept below 200°C (time 150 s). Dispersing or wetting agents (especially ethylene oxide condensates) increase the amount of disperse dyes fixed and respectively allow the time or temperature to be reduced without decreasing the amount of dye taken up. Rate of diffusion of disperse dyes in the fibre is high and unaffected by dispersing agent, but these substances facilitate the entry of the dye into the outer layer of the fibre, thus setting up the concentration gradient necessary for diffusion to occur. W.M.

### Dyeing Properties of Polyacrylonitrile Fibres with Special Reference to New Dralon

W. Hees

*Melliand Textilber.*, 42 (Jan 1961) 87–88

The affinity of polyacrylonitrile fibres for basic dyes results from the presence of acid groups introduced by the catalyst (NH<sub>4</sub> persulphate) and the activator (sodium bisulphite) during polymerisation. Saturation values of basic dyes, which are proportional to the number of acid groups, should be high enough to permit the dyeing of deep shades. New Dralon is a fibre with increased saturation value for basic dyes (55 millimol compared with 37 millimol). Deep blacks can be obtained with Astrazon Black WRL. In mixtures of New Dralon with other fibres (cellulosic fibres, wool) the high affinity of New Dralon for basic dyes reduces staining of the other fibre to a minimum. W.M.

### Mechanism of Dyeing of Acid Dyes on different Polyamide Fibres

H. Zollinger, G. Baek, B. Milioevic, and A. Roseira

*Melliand Textilber.*, 42 (Jan 1961) 73–80

There is a close relation between the number of terminal NH<sub>2</sub> groups in a polyamide fibre and the amount of acid

dye taken up. The salt linkage between the anionic dye and the cationic groups in the fibre can be formulated by assuming either un-ionised amino acids ( $\text{NH}_2 \dots \text{COOH}$ ) or amphoteric ions ( $^+\text{NH}_3 \dots \text{COO}^-$ ). If thermodynamical treatment is applied to absorption equilibria, no term for number of carboxylic acid groups appears in the equation in case of un-ionised amino acids but does in the case of amphoteric ions. Experiments to decide between the two possibilities by comparing the dyeing properties of normal nylon 6.6 with those of a specially produced polyamide with double the number of carboxylic groups but the same number of  $\text{NH}_2$  groups seem to indicate the presence of amphoteric ions but the results are not considered to be conclusive. Efforts to distinguish between the two possibilities by finding out which equation fits the experimental measurements best were unsuccessful and it is concluded that dyeing experiments cannot give the answer. Dyeing of polyamide fibres with acid dyes takes place partly by salt linkage between dye and fibre and partly by solution of the dye in the fibre. The tendency of a dye to form aggregates is related to its solubility in polyamide fibres.

W.M.

#### Influence of Dry and Wet Heat Setting on the Dyeing Properties of Nylon 6 Fibre

T. Yasuda

J. Soc. Textile Cellulose Ind. Japan, 17 (Jan 1961) 55-59

Fabrics processed by the wet heat-setting technique absorb more dye than fabrics subjected to dry heat-setting, and the rate of diffusion of dye into the fabric is also greater, even when the materials are of equal density. This is considered to be due to a growth of crystallites and increase in inter-crystallite distance taking place on wet heat-setting, whereas dry heat-setting results in an increase in the number of crystallites rather than in their volume, accompanied by a reduction in the distance between the crystallites.

I.G.L.

#### Colouring Chlorine-containing Fibres

M. Gord

Tintex, 26 (15 Feb 1961) 107-125

The chemical and physical characteristics of these fibres, e.g. Rhovyl (I), Thermovyl (II), Fibravyl (III) and Retraetyl 30 (IV) are fully described with special reference to general and individual variations in shrinkage in water (e.g. in  $\text{H}_2\text{O}$  at  $95^\circ\text{C}$ , I and II shrank 55%, III 30%, and IV nil) and how this is affected by time and temperature and fibre tension (e.g. in  $\text{H}_2\text{O}$  at  $95^\circ\text{C}$  I-continuous filament has 52.4% retraction at tension 0 g/den, 10.8% at 0.04 g/den, and breakage at 0.17 g/den). Choice of dyes is mainly limited to indigosols, azoic combinations and, especially, plastosoluble (disperse) dyes. The last-named tend to have excellent wet fastness but poor light fastness. A list of 103 commercial disperse dyes of light fastness 5 and over is given. Carriers are generally necessary and dyeing is said to occur through solution of dye in carrier. The physical and chemical requirements of suitable carriers are described, and the two recommended, Solvant FT and *o*-phenylphenol, are discussed in detail. A.T.P.

#### PATENTS

##### Fluorescent Brightening of Cellulosic Materials

Ciba

BP 862,645 (Switzerland 16 Mar 1956)

The material is treated with a powdered fibre-forming polymer of particle size  $< 50\mu$  carrying on it 0.01-5.0% of its weight of a fluorescent brightening agent containing at least one azole ring. Thus nylon is dissolved in 96%  $\text{H}_2\text{SO}_4$ , poured into ice water and the precipitate washed free of acid and dried. The powder obtained is stirred for 30 min in a bath made up of water (3000), 86%  $\text{HCOOH}$  (1),  $\alpha\beta$ -di-(5-methylbenzoxazolyl)-(2)-ethylene (0.24), and a non-ionic dispersing agent. The treated powder (15) is filtered off, dried and ground for 48 h, with a condensate (7.5) of octadecyl alcohol and 30-40 ml ethylene oxide and water (77.5). The product is used as a fluorescent brightening agent for adding to viscose spinning solutions.

C.O.C.

##### Dispersing a Colorant in Viscose

American Enka Corp.

USP 2,934,448 (19 May 1954)

The colorant is added to a branch stream of the viscose which is then introduced into the main stream. The dispersed colorant is added to the branch stream through a special T-connection having a nozzle which ensures that all the dispersion is introduced coaxially or centrally of the short stream and in a direction toward the high capacity pump. This pump is one of two in the branch stream, the

second being of higher capacity than the other, thus creating a suction zone in the stream. This system results in good homogeneous mixing without need for additional elaborate equipment.

C.O.C.

##### Pigmented Viscose Rayon

American Enka Corp.

USP 2,934,440 (24 May 1957)

A colorant is added to viscose in amount sufficient to impart a full colour to the spun filaments and sufficient of an aqueous suspension of an inorganic pigment to prevent dichroism but insufficient to cause delustring. The viscose is then extruded into a suitable coagulating bath. Thus use of Monastral Fast Green (C.I. Pigment Green 7), Monastral Fast Blue (C.I. Pigment Blue 15),  $\text{TiO}_2$  and Bentonite (C.I. Pigment White 6 and 19) (0.140, 0.075, 0.050, and 0.019 respectively on weight of cellulose) yields filaments which do not vary in colour when viewed at different angles.

C.O.C.

##### Fixing Pigments on Fibrous Materials

FH

USP 2,933,416 (26 Apr 1955)

Pigments are fixed fast to washing by applying them in aqueous alkaline solution or dispersion together with an alkali-soluble resinous condensate containing reactive  $\text{COOH}$  groups and then heat curing. The condensate is obtained by treating a polybasic acid with a polyhydric alcohol and a polyfunctional compound whose functional groups contain a three-membered ring containing a N or O atom.

C.O.C.

##### Chemical Modification and Dyeing of Keratinous Materials

U.S. Secretary of Agriculture

USP 2,933,365 (20 Nov 1957)

Reduced keratin is treated with a compound containing an acyl-N group linked through the N to an aromatic group. The acyl radical contains an olefinic double bond between two C atoms, at least one of these bonds being directly linked to the non-oxo carbonyl group of the acyl-N group. The aromatic group contains at least one chromophore. If the compound contains only one olefinic double bond the keratin is only dyed, whereas if it contains more than one olefinic double bond both dyeing and cross-linking of the polypeptide chains result. This yields dyeings of excellent fastness to washing. Thus wool yarn was reduced with 2-mercaptoethanol and then treated with *N*-(*p*-phenylazo-phenyl)maleimide dissolved in 2-methoxyethanol. This yielded a yellow dyeing of good fastness to washing.

C.O.C.

##### Improving the Brightness of Dyeings of Metallised Dyes

Whiffen &amp; Sons

BP 859,279 (26 Mar 1958)

Dyeings of metallised dyes on cellulose, wool, or synthetic-polymer fibres are brightened by treatment with an aqueous solution of a sequestering agent. Thus wool dyed with Irgalan Orange RL (C.I. Acid Orange 86) treated for 15-30 min at  $30^\circ\text{C}$  in a bath containing Lissapol NC (1 g/l.), sodium ethylenediamine tetra-acetate (2) and 85% formic acid (4 ml/l.) at a liquor ratio of 50:1, had its colour much brightened with insignificant loss in depth or fastness.

C.O.C.

##### Dyeing Polyester Fibre

DuP

USP 2,934,397 (26 Oct 1955)

Dimethyl terephthalate is an excellent carrier for use in boiling aqueous disperse dye baths. It is neither toxic nor malodorous and presence of  $< 0.2\%$  in the liquor enables production of level, heavy dyeings of good fastness to light.

C.O.C.

##### Interactions of Surface-active Agents with Congo Red (III p. 257)

##### Colour Effects Produced in Nearly White Fabrics by Household Detergents (VII p. 266)

##### Vinyl Polymers containing a Chemically Condensed Azoic Coupling Component (XIII p. 273)

##### Interfacial Tension and Textiles (XV p. 276)

## IX—PRINTING

### Reproduction of Mode Shades in Printing with Reactive Dyes

A. Schaub

S.V.F. Fachorgan, 16 (Jan 1961) 56-58

A survey of the troubles which can occur in printing with reactive dyes, i.e. those due to the water used and its pH, standing time of the paste, copper sensitivity, drying

temperature, urea content, etc., and methods of avoiding some of these troubles. P.B.S.

## PATENTS

### Transfer of Coloured Pictures to Printing Surfaces

Dr. Bekk & Kaulen Chemische Fabrik

BP 859,817 (Germany 22 Sept 1956)

Separate copies of the picture, one for each colour, are prepared on transparent sheets. These are then reproduced, either by drawing or photography, on transparent plastic foil and from these are transferred directly on to the printing surface, e.g. a photogravure roller for textile printing, a screen or hatching having been copied in during one of the photographic processes. C.O.C.

### Dextrans as Thickeners for Printing Pastes

Commonwealth Engineering Co.

USP 2,933,403 (13 Feb 1957)

Highly adhesive, water-soluble native dextrans are unsuitable as thickeners, but the short tough gums of viscosity 1.16–0.57 cP in water at 25°C and the crumbly short gums of viscosity 1.36–0.50 cP in water at 25°C are very satisfactory thickeners, particularly for photogravure printing of textiles. The tough gums are obtained by the action of certain strains of *Leuconostoc mesenteroides* on sucrose while other strains yield the crumbly gums. C.O.C.

### Discharge Prints on Polyester Fibres

FH

BP 859,015 (Germany 8 Dec 1956)

White or illuminated discharge prints on polyester fibres are obtained by printing on to a dyed ground a paste containing a reducing agent and anthraquinone and, in the case of illuminated discharges, a vat dye. C.O.C.

### Blue Prints

Horizon

USP 2,935,403 (25 Apr 1958)

A substrate, preferably a sheet of anodised aluminium, is sensitised with a photolytically reducible compound, e.g. ferric ammonium oxalate. It is then illuminated through a stencil and the exposed sheet treated for 10–30 s with dil. aq. K ferrocyanide. This yields a pale blue image which, on the sheet being dipped in 0.1–0.5%  $H_2O_2$ , becomes an intense Prussian Blue. The faint bluish tint in the unexposed background is removed by treating at the boil with distilled water for 30 min which also seals the anodised layer. C.O.C.

### Yellow Dye Images by Colour Development

General Aniline

BP 865,032 (U.S.A. 3 Apr 1958)

Yellow images of excellent fastness can be produced simultaneously with cyan and magenta azine dye images by colour-forming development with 2,4-diaminoaniline in presence of 1-benzoyl-5-amino-2-naphthol. C.O.C.

Solutions of Azo Pigments (IV p. 260)

Fixing Pigments on Fibrous Materials (VIII p. 268)

Tiled Effect on Linoleum or the like (X p. 270)

Improving the Printability of Polyethylene (XIII p. 273)

Interfacial Tension and Textiles (XV p. 276)

## X—SIZING AND FINISHING

### A Simple Explanation of Drying Theory for Textile Technologists

J. Stipek

S.V.F. Fachorgan, 16 (Jan 1961) 2–29

### Convection Driers and Determination of Their Performance

K. Prehn

Ibid., 30–46

### Theoretical Considerations of Cellulose Cross-linking

J. L. Gardon and R. Steele

Text. Research J., 31 (Feb 1961) 160–171

The hypothesis that covalent cross-linking is the best way to improve the resilience of cellulosic fabrics is supported by much available chemical evidence. The theories relating physical properties to cross-linking in rubbery polymers are reviewed and it is shown that they apply in some qualitative aspects to cellulose. In particular, observed changes in swelling, solubility, modulus, tensile strength, and resilience are consistent with these theories. The concentration of intermolecular cross-links was estimated on the basis of published data and it was found that, to attain maximum crease-recovery value, at least every twenty-fifth accessible anhydroglucose unit has to

be intermolecularly cross-linked. In rubbery polymers substantial changes in mechanical properties occur at much lower levels of cross-linking. This difference between cellulose and rubbers in response to cross-linking is probably attributable to the polar nature of cellulose. 52 References. C.O.C.

### Application of the APO-THPC Flame Retardant to Cotton Fabric

G. L. Drake, J. V. Beninate, and J. D. Guthrie

Amer. Dyestuff Rep., 50 (20 Feb 1961) 129–134

A durable flame-retardant treatment has been developed which consists in treating cotton fabric with a mixture of tris(1-aziridinyl)phosphine oxide (APO) and tetrakis-(hydroxymethyl)phosphonium chloride (THPC), drying and curing to form an insoluble resin inside the fibre, e.g. an 8.5 oz/yd<sup>2</sup> cotton sateen is padded in 18.5% Imine IP (an 85% commercial solution of APO), 17.3% THPC, 4.3% triethanolamine, and 0.5% Triton X-100 (wetting agent). The temperature of the mixture is kept below 70°F by addition of ice, the wet pick-up on padding is 58%, the drying temperature 185°F, and the curing conditions 3 min at 345°F, followed by washing, drying, and softening. About 10% of resin solids is required to give durable flame resistance. W.G.C.

### Mechanism of the Loss of Copper from Cotton Fabric during Actinic Breakdown under Conditions of Wetting. I—Preliminary Experiments with Copper Carbonate

G. R. F. Rose, J. B. Clifford, and C. H. Bayley

Text. Research J., 31 (Jan 1961) 1–14

The factors responsible for the rapid loss of most fungicides from cotton cloth exposed out of doors include the water-solubility of the fungicide, actinic breakdown of the fungicide or the cotton, reaction between the fungicide and the cotton, and mechanical loss of the fungicide. Intermittent leaching and drying of bleached cotton containing copper carbonate causes more breakdown and loss of copper than does continuous leaching. Similarly, by exposure to light containing moderate amounts of radiation of 3000–4000 Å, presence of copper increases the amount of deterioration. Loss of copper caused by leaching following exposure, or by intermittent exposure and leaching, increased with increased exposure to light. Exposure to light with intermittent dampening with water, with or without drying before re-exposure, increased the tendency of the copper to leach out. There was marked migration of copper in these experiments. The loss of copper appeared to involve solubilisation of the copper by oxidation products of cellulose. C.O.C.

### Chemical Shrink-resistant Finishing of Cotton Fabrics

F. Nestelberger

Textil-Rund., 16 (Feb 1961) 55–63

A series of experiments (30) is described in which the effects of synthetic resin treatments of cotton cloth are related to shrink-resistance. The resins are all condensates of formaldehyde with urea, substituted ureas, or melamin, which are condensed on the fabric by heating, with magnesium chloride as catalyst. The latter half of the experiments show the effects of the addition of starch and modified starches to the simple resin treatments. P.B.S.

### Attachment of Active Hydrogen Compounds to Cellulose with Divinyl Sulphone

C. M. Welch and J. D. Guthrie

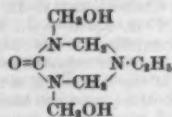
Text. Research J., 31 (Jan 1961) 84–86

Divinyl sulphone reacts readily with cotton and rayon, both vinyl groups of the sulphone adding to form cross-linked cellulose ethers— $CH_2CHSO_2CH_2CH_2 + 2 \text{ Cell-OH} \xrightarrow{OH^-} \text{Cell-O-CH}_2CHSO_2CH_2CH_2\text{-O-Cell}$ . However, in presence of amines, alcohols, amides, phenols, ketimines and sulphur compounds, the reaction can proceed so that only one vinyl group is attached to the cellulose and the other to an active hydrogen compound, e.g.  $R^1R^2NH + CH_2CHSO_2CH_2CH_2 + \text{Cell-OH} \xrightarrow{OH^-} R^1R^2NCH_2CHSO_2CH_2CH_2\text{-O-Cell}$ . By this means such compounds, even large dye molecules, have been attached to cellulose. Irrespective of the type of compound used, the treated cottons were insoluble in cupriethylenediamine showing that cross-linking had occurred with substitution. In some cases high wet crease recovery and, more rarely, high dry and wet crease recovery were obtained. C.O.C.

**Reactive Finishing Agents for Cellulose**

L.-H. Marcon *Textiles*, 26 (15 Feb 1961) 87-105

General survey of "reactive" finishes, i.e. production of "wash and wear" fabrics. The reactivity of OH groups in cellulose permits the use of a wide range of finishing agents, e.g.  $-NCH_2OH$  compounds, acetals, ethyleneamines, cyclopropanes, pyridinium salts. Of these, the first is the most important and is fully discussed with comparison of various chemical structures with, e.g., rubbing resistance, % resin lost on washing. Stability of finishes to chlorine is described. Thus,  $OCl_2$  reacts with methylolamides to give chloramines (I) ( $-CO-N-R-Cl$ ) which are stable to washing and unaffected by bisulphite and perborate. The formation of (I) is often indicated by a slight odour of  $Cl_2$  or a yellowing of the fabric. They decompose on heating to give nascent oxygen and HCl which cause fibre degradation. Absorption of  $Cl_2$  and resultant fibre weakening are compared for various compounds. However, although e.g. trimethylolmelamines retain more  $Cl_2$  than dimethylol urea, fibre degradation is more drastic with the latter. Stability to  $Cl_2$  is then related to chemical structure and to the extent of condensation of finishing agent with the fibre. Thus if condensation is incomplete, residual  $-NCH_2OH$  groups may eliminate  $HCHO$ , leaving  $-NH$  susceptible to  $Cl_2$  absorption. The extent of condensation is related to catalyst, time and temperature of condensation. Several newer polymethylol derivatives are described, principally triazinones



and structures based on 1,3-bis(hydroxymethyl)imidazoline-2. A recommended formula is a triazinone or ethylene urea- $HCHO$  condensate (120-150 g/l.),  $MgCl_2$  (20 g/l.), polyethylene emulsion (30-50 g/l.)—to increase abrasion resistance—softening agent (2-10 g/l.), and waterproofing agent (40-50 g/l.), padded to 70% pick-up, treated at 160°C for 4 min and soaped at 50-60°C in presence of traces of perborate. 54 References. A.T.P.

**Water-repellent Treatments with Silicone and Paraffin Wax Emulsions**

U. Rall *Melliand Textilber.*, 42 (Feb 1961) 212-215

Experiments to compare effect of silicone and paraffin wax emulsions showed that on cotton the two emulsions give equally good water repellency, that in both cases combination with pre-condensates of the melamine types improves results still further, and that the better handle is obtained with silicones. On wool and silk, excellent results can be achieved only with silicones. On synthetic fibres silicone emulsions give an excellent water-repellent finish. Perlon and Trevira fabrics treated with paraffin emulsions showed a water uptake (Bundemann test) of about 20%. On Dralon paraffin emulsions have no effect. W.M.

**Felting Investigations. II—Relationship between Crimp and Rate of Felting**

W. G. Crewther and L. M. Dowling *Text. Research J.*, 31 (Jan 1961) 14-18

Removal of crimp facilitates felting, whereas imposition of tight coils in the fibres retards felting. C.O.C.

**PATENTS****Simultaneously Dry Cleaning and Imparting Water-repellency to Garments**

Commonwealth Engineering Co. *USP* 2,933,411 (18 June 1953)

During dry cleaning the goods are impregnated or coated with a dextran ester of a saturated fatty acid of 8-18 C and containing 2-3 fatty-acid radicals per anhydroglucopyranosidic unit. C.O.C.

**Imparting Flame-resistance to Cellulose Ether Textiles by Treatment with Aziridinyl Phosphine Oxide or Sulphide**

U.S. Secretary of Agriculture *USP* 2,933,367 (29 Oct 1957)

Treatment with a compound containing two or more

1-aziridinyl  $\left( \begin{array}{c} \text{CH}_2 \\ | \\ \text{N} \\ | \\ \text{CH}_2 \end{array} \right)$  groups directly attached to P atoms is used. Thus phosphonomethylated cotton sheeting (obtained by treating cotton with chloromethyl phosphonic acid in presence of a strong base) was soaked in 3% HCl for 1 h, rinsed free of HCl and air dried. It was then treated with tris(1-aziridinyl)phosphine oxide and cured for 1 min at 450°F. The treated fabric had good crease recovery and flame resistance. C.O.C.

**Reducing Felting and Shrinking Powers of Wool Textiles**

U.S. Secretary of Agriculture *USP* 2,933,366 (12 May 1958)

The material is impregnated with a polyepoxide and a polyamide and then treated with gaseous ammonia. This yields a shrink-resisting and crease-shedding finish without affecting handle, resiliency, porosity, tensile strength, and colour. C.O.C.

**Setting Keratinous Fibres with Mercaptoalkanoic Acid Amides**

Société Monsavon-l'Oreal *BP* 859,347 (France 29 May 1958)

The fibres are impregnated with a freshly prepared aqueous solution obtained by adding to an aqueous stock solution at pH 8.0-9.5 of an *N*-monosubst.- or *N*-unsubst.-mercaptoalkanoic acid amide, an acid or acidic buffer so as to bring the resulting solution to pH 5.0-7.5. The fibres are then brought into the desired formation and then set by a known method. The stock solutions are stable while at pH 8.0-9.5 and when the pH is lowered for use, decomposition of the amide is too slow to have any material effect. C.O.C.

**Permanent Creasing**

U.S. Secretary of Agriculture *USP* 2,933,409 (14 Aug 1958)

The material is impregnated with a polyamide and a polyepoxide, creased or pleated, and then heated to cure the polymers, e.g. in a Hoffman press. This yields creases resistant to long wearing, washing, and dry cleaning. The treatment also confers shrink-resisting and crease-recovery properties. C.O.C.

**Treating Yarns and Fabrics of Man-made Fibres to render their Structures More Stable**

H. R. Hilfiker & Co. *BP* 859,574 (Switzerland 30 May 1956)

Materials made of fully set, multi-filament yarns are treated in an aqueous bath containing a shrinking agent and simultaneously subjected to ultrasonic vibration. Thus nylon stockings are successively treated in three baths, the first containing an aqueous solution of phenol, the second an aqueous solution of caustic soda and the third water containing 1 g/l. of triethanolamine as a wetting agent. The first two baths are exposed to ultrasonic radiation of frequency 20,000-60,000 cycles/s. C.O.C.

**Improving the Stability to Heat of Polyacrylonitrile Fibres**

Courtaulds *BP* 864,312 (14 Apr 1958)

Polyacrylonitrile fibres, particularly those containing carboxylic acid groups, when heated at < 85°C in an aqueous solution containing < 0.05% of an ammonium or alkali-metal salt of an aliphatic carboxylic acid having reducing properties, e.g. Na formate and K oxalate, have improved stability to heat. C.O.C.

**Fire-resistant Cloth**

Lindsay & Williams *BP* 859,502 (25 Feb 1958)

A textile base is impregnated or coated with a composition comprising polychloroprene,  $Sb_2O_3$ , and either chlorinated paraffin wax or chlorinated diphenyl. A pigment may also form part of the composition. The product is used as a fire-resistant covering on electric cables and as a brattice cloth. C.O.C.

**Tiled Effect on Linoleum or the like**

Congoleum-Naim *USP* 2,935,417 (17 Apr 1956)

A fibrous backing is coated with paint which is then allowed to set. Narrow strips are then painted on it with a paint, part of whose binder consists of a tempering agent obtained by adding to a liquid, heat-bodied drying oil a basic substance which reacts with the free acid portion of the oil. This yields sharply defined, depressed prints resembling the mortar lines between tiles. C.O.C.

### Coating a Backing with a Thermoplastic Composition

Armstrong Cork Co. BP 862,466 (U.S.A. 13 Mar 1958)

The powdered or granulated composition is applied while the backing is continuously moving in one direction. The layer of composition is then softened and consolidated by being passed through a nip formed by a non-yielding roll and a resilient roll.

C.O.C.

### Embossing and Venting Thermoplastic Resin-coated Fabrics

DuP BP 859,963 (U.S.A. 24 May 1957)

A moving wire mesh belt is continuously pressed into and through the coating previously softened by heat. The pressure is maintained for  $< 10$  s and then the belt is continuously separated from the fabric. This yields an embossed vented resin-coated fabric in which the vents are narrower at the surface of the coating than at a deeper level. This yields a fabric of excellent pliability, surface appearance, and permeability to water vapour.

C.O.C.

Water-repellent and Rot-proofing Composition (III p. 258)  
Vinyl Phosphoric Acid and its Derivatives—Flame-retarding Agents (III p. 258)

Cyanooethylation of Cotton in Aqueous Medium (VI p. 263)  
Crystallographic Changes in Wool Keratin during Chemical Modification, especially Finishing (VI p. 264)

Irradiation of Nylon (VI p. 266)

Influence of Dry and Wet Heat Setting on the Dyeing Properties of Nylon 6 Fibre (VIII p. 268)

Dimensional Stabilisation of Cellulose Materials (XI p. 272)

Acid Hydrolysis of Dimethylolurea Polymerised in Cotton (XIV p. 275)

Interfacial Tension and Textiles (XV p. 276)

## XI—PAPER AND OTHER CELLULOSIC PRODUCTS

### Alkaline Degradation of Glucose and of some of its Acetyl Derivatives

W. M. Corbett and A. M. Liddle J.C.S., (Feb 1961) 531-538

In soln. of OH-ion concn. 0.01-0.05 g-ions/l., the yield of total acid from glucose is inversely related to the OH-ion concn., being 2.5-1.5 equiv., and increase in acidity is mainly due to formation of formic acid; glucosaccharinic is the main saccharinic acid except at high concn. of Ca ion, when yield of glucosaccharinic acid becomes comparable. A brief study is also reported of the degradation of tetra-*O*-acetyl-2-hydroxy-D-glucol and the early degradation stages of penta-*O*-acetyl-D-glucose, 2,3,4,6-tetra-*O*-acetyl-D-glucose, and penta-*O*-acetyl-aldehyde-D-glucose.

H.H.H.

### Degradative Action of Hot Dilute Alkali on Hydrocelluloses

R. L. Colbran and G. F. Davidson

J. Textile Inst., 52 (Feb 1961) T73-T87

When hydrocellulose is heated at 100°C with dilute alkali it suffers a loss in weight, owing to formation of soluble degradation products, that increases with time but at a diminishing rate, becoming almost zero after a limited time of heating. The insoluble residue shows a progressive fall in reducing power and rise in carboxyl content. The ultimate loss in weight, reducing power, and carboxyl content depend on the nature and concentration of the alkali. The ultimate weight loss is much greater with sodium or potassium hydroxide than with hydroxides of alkaline-earth metals. With strontium and barium hydroxides it decreases with increasing alkali concentration in the range 0.1-0.4  $\%$  and is generally less with strontium than with barium hydroxide. The ultimate weight loss rises, however, with increasing concentration of sodium hydroxide in this range. Use of 0.4- $\%$  strontium hydroxide results in almost complete elimination of reducing power, and the carboxyl content is higher and weight loss lower than with any other reagent used. The reaction between sodium hydroxide and hydrocellulose comes virtually to a stop while the reducing power is still very considerable and the carboxyl content much lower than when 0.4- $\%$  strontium hydroxide is used. Some theoretical aspects are discussed.

W.R.M.

### Amorphous Cellulose

V. I. Sharkov and V. P. Levanova

Zhur. priklad. khim., 33 (Nov 1960) 2563-2571

Natural cellulose is made amorphous not only by mechanical grinding, but also by high compression or by

passing it between rollers. Amorphous cellulose is very sensitive to water or moisture, which increases its density and the orientation of the macromolecules and decreases its internal surface. Density decrease of natural cellulose during its transformation into the amorphous state is analogous to decrease in density of crystalline glucose during its change into a glass-like form. Natural cellulose has high heat capacity which is greatly decreased by mercerisation and dry grinding. A comparison of properties of ground and natural cellulose suggests that the latter contains a large proportion of highly oriented or crystalline regions, in contrast to the former, which has a typically amorphous structure.

T.Z.W.

### Properties and Nature of the Surface of Cellulose. III—Cellulose in contact with Organic Liquids and Aqueous Solutions of Reduced Surface Tension

K. Borgin

Norsk Skogindustri, 14 (Nov 1960) 485-495 (in English)

The angle of contact of organic liquids with hydrophilic groups is greatly influenced by the r.h. of the air and by the moisture content of the cellulose. The contact angle of glycerol and that of ethylene glycol decrease with increasing moisture content up to 12% water absorption, after which they increase, which is taken as further proof that regenerated cellulose binds 12% water as a solid hydrate. The contact angles of liquids which do not contain hydrophilic groups and which spread completely on water are independent of water content, while liquids that spread only partially have angles which increase with the water content of the cellulose. Decreasing the surface tension of the water increases the capillary wetting of hydrophobic solid materials. Since cellulose is strongly hydrophilic, any decrease in surface tension will have a negative action on capillary wetting.

R.A.

### Influence of pH on the Oxidation of Cellulose with Chlorine Water

N. E. Virkola and O. Lehtikoiki

Paperi ja Puu, 42 (Nov 1960) 559-564 (in English)

Max. values for chlorine-consumption and for carbonyl and carboxyl content are obtained at pH 2 and 7, respectively, the corresponding min. values being at pH 1 and pH 4-5. There is a linear relation between chlorine consumption and degree of oxidation. The degree of yellowing was approx. the same for samples oxidised at pH 2-5 and at pH 6, while samples oxidised at pH 4 yellowed less. Reduction with NaBH<sub>4</sub> reduced yellowing to the same level for all pH values.

R.A.

### Effect of Bleaching Conditions on Greaseproof Properties of Sulphite Pulp

J. Protekta Przegląd Papierniczy, No. 10 (1959) 299-295

Polish Tech. Abs., No. 2 (38) (1960) 141

An investigation of the influence of amount and manner of chlorine addition, type and amount of agent used in intermediate washing, bleaching agent, temp. and pH of final stage. Results are evaluated in terms of the "product of greaseproof property", degree of whiteness, yield, viscosity, and  $\beta$ - and  $\gamma$ -cellulose content. An attempt is made to correlate content of low molecular carbohydrates and pulp properties, and a modified bleaching method is described giving pulps of low "product of greaseproof property".

R.A.

### Fluorescent Brightening of Paper

S. Bartkiewicz

Przegląd Papierniczy, No. 9 (1959) 257-260

Polish Tech. Abs., No. 2 (38) (1960) 139, 141

The effects of Loucophor BS, Blankophor BUP, Heliophor BB, and Fluolite C on wood-free paper bleached in the beater, and the effect of Blankophor BUP on paper coated with a mixture of china clay and starch have been studied, using a Pulfrich photometer with an Ulbricht ball. Best results with bleaching in the beater were obtained with Loucophor BS and Blankophor BUP, followed by Fluolite C > Heliophor B. Blankophor BUP in coating mixtures was satisfactory but not as good as with bleaching in the beater.

R.A.

### Fluorescent Brightening Agents and their Behaviour in the Paper Industry

A. J. Hinton

Textil-Rund., 15 (Dec 1960) 654-658

Disubstituted diamino disulphostilbenes are the most commonly used brighteners in the paper industry. If added in the hollander the brightening effect is determined

by the degree of bleaching, quality of the size, rosin, and water used. White water should only be used with acid-resistant brighteners. The effect of sizing and acidity has been studied for 16 commercial (mainly unnamed) brighteners. Most red-toned, but few green-toned, fluorescers work satisfactorily with waste water. R.A.

#### Tables of Light-scattering Coefficients from Reflectance Data

E. B. Koller and S. M. Chapman

*Svensk Papperstidning*, 64 (28 Feb 1961) 113-124 (in English)

If the reflectances of a sheet of paper of given basis weight are known, the reflectances and printing opacity of a sheet of similar character but different basis weight may be determined by means of the Kubelka-Munk equation. The calculations are, however, tedious. Tables are therefore presented which greatly simplify the calculations. M.T.

#### PATENTS

##### Fillers and Pigments for Paper

R. T. Vanderbilt Co. USP 2,935,438 (2 Mar 1956)

Finely divided, precipitated hydrated Ca silicate, which has not been dried after precipitation, is treated with Al sulphate in aqueous medium at a concentration of < 1% so that < 50% of the Ca silicate is not in solution. Preferably the reaction is carried out under conditions of intense agitation or shear. The insoluble precipitate, when used without drying, is an excellent filler or pigment for adding to paper during its manufacture, e.g. in the pulp slurry or in a coating composition. It provides a cheap way of improving the physical and optical properties of paper. C.O.C.

##### Sizing of Paper

American Cyanamid Co.

BP 864,336 (U.S.A. 17 June 1958)

Improved wet resistance and dry strength are obtained without increasing the wet strength by addition to the pulp of 0.05-3% (based on the dry wt.) of a water-sol. cationic vinyl polymer formed by quaternisation of a chlorinated *ar*-methyl styrene with a water-sol. tertiary amine, e.g. pyridine. R.A.

##### Paper having Improved Retention of Pigments and Dyes

National Starch & Chemical Corp.

USP 2,935,436 (9 May 1957)

Incorporation into the paper during its manufacture of a tertiary aminoalkylated starch ether results in a paper of greater strength, improved retention of pigments and dyes, and better finish. C.O.C.

##### Pigment-filled Paper

Columbia-Southern Chemical Corp.

USP 2,935,437 (20 Nov 1953)

Paper of high brightness, opacity, and smoothness is obtained by adding a finely divided, hydrated amorphous Ca or other alkaline-earth-metal silicate to wood pulp slurry kept at pH 4.0-9.2 throughout the addition by means of an acidic substance which forms a water-insoluble salt with the alkaline-earth silicate, e.g. Al sulphate. The resulting paper has the same brightness, opacity and bursting strength as paper pigmented with  $\text{TiO}_2$  (C.I. Pigment White 6). C.O.C.

##### Protein Compositions for Coating Paper

Borden Co.

USP 2,933,406 (11 Sept 1957)

A combination of alkali-soluble protein, e.g. casein, and a non-ionic surfactant yields a coating which is free from pinholes and surface roughness. The surfactant releases any air retained in the protein solution. The surfactant must be soluble in water and ether, and it is mixed with the protein until it becomes extractable with difficulty, if at all, from the mixture by ether. C.O.C.

##### Dimensional Stabilisation of Cellulose Materials

Upson Co.

BP 860,025 (U.S.A. 10 Aug 1956)

Cellulose materials are rendered dimensionally unaffected by change in humidity by treating them with an acid-type stabiliser, cf. BP 846,098 and 849,522 (J.S.D.C., 76 (1960) 710; 77 (1961) 85), which has at least partly reacted with a basic organic compound containing > 1 amino group. Thus unsized cellulose fibre board, treated with an aqueous solution containing ethylenediamine and adipic acid in equimolecular amounts so that the

treated board contained 35-3% of the resulting salt, lost 68.8% of its normal hygroexpansivity when exposed to r.h. change of 0-90%. C.O.C.

##### Fluorescent Brightening of Cellulosic Materials (VIII p. 268)

##### Coating a Backing with a Thermoplastic Composition (X p. 271)

##### Comparison of Kappa Number Test with other Methods for Determining Degree of Cooking of Chemical and Semi-chemical Pulps (XIV p. 275)

## XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

### Synthetic Polypeptides and Proteins

C. H. Bamford

*J. Textile Inst.*, 51 (Dec 1960) T1527-T1537

Evening Discourse presented during the Second Quinquennial International Wool Textile Research Conference in May 1960. Primarily a discussion of protein structure. M.T.

### Structure of Globular Proteins

J. C. Kendrew

*J. Textile Inst.*, 51 (Dec 1960) T1517-T1519

Summary of an Evening Discourse presented during the Second Quinquennial Wool Textile Research Conference in May 1960. M.T.

#### PATENTS

##### Tanning Agents from Ammonium Bisulphite and Phenol-Formaldehyde Condensates

FBY

BP 864,079 (26 Nov 1958)

Good-quality leather is obtained by tanning with the product obtained by treating a low mol. wt. phenol-formaldehyde condensate containing methylol groups with  $\text{NH}_4$  bisulphite. C.O.C.

##### Resinous Tanning Agents

FBY

BP 863,505 (Germany 23 July 1958)

Mineral tanned leather is improved by treatment with an aqueous solution of the mixed condensates or with their alkali-metal,  $\text{NH}_4$  or amine salts, obtained from aromatic hydrocarbons containing condensed benzene nuclei, HCHO and aromatic carboxylic acids containing at least one OH group capable of reacting with HCHO. The process is particularly valuable for the retanning of chrome leather to obtain plumping. C.O.C.

##### Siloxane Compositions for rendering Leather Water-repellent

Midland Silicones

BP 862,469 (U.S.A. 29 May 1958)

The resistance to water penetration of leather is increased from five- to one hundred-fold by treatment with a mixture of (1) 15-50% by weight of a compound  $\text{CH}_3\text{Si}(\text{OR}^1)_2$  ( $\text{R}^1 = \text{Alk}$  or acyl of 1-4 C), (2) 50-70% of a methylpolysiloxane resin composed of trimethylsiloxy and  $\text{SiO}_2$  units, the ratio of  $\text{CH}_3:\text{Si}$  being 1:1 to 2:1, and (3) 15-80% by weight of a polysiloxane  $\text{R}^2\text{SiO}_{1-n}$  (each  $\text{R}^2 = \text{Alk}$  or alkenyl of < 4 C or monocyclic Ar;  $n = \text{on average } 2.0-2.9$ ). C.O.C.

##### Washable, Glossy, and Non-staining Leather by use of Organopolysiloxanes

Société des Usines Chimiques Rhône-Poulenc

BP 863,771 (France 12 May 1958)

The leather is coated with a composition comprising (1) 4-30% by weight of a methylphenylpolysiloxane in which  $(\text{CH}_3 + \text{C}_6\text{H}_5):\text{Si} = 1.3-1.6$ , and  $\text{C}_6\text{H}_5:\text{Si} = 0.4-0.7$ , 1-5% hydroxyl groups are attached to Si and a 70% solution in toluene has viscosity < 120 cS at 20°C, (2) 1-20% of a wholly organic resin drying to a thin glossy film, and (3) 50-95% of a volatile solvent. An example is Rhovinal B10 (polyvinyl butyral) (9g), xylene (40-5), isopropanol (40-5), and Rhodorsil 6407 (70%) (polysiloxane) (10g). C.O.C.

## XIII—RUBBER; RESINS; PLASTICS

### Carbon Black Masterbatching Processes

E. M. Dannerberg, E. Hagopian, J. P. Hall, and A. I. Medalia

*Trans. I.R.I.*, 37 (Feb 1961) 1-19

Masterbatches of rubber with carbon black can be obtained by mixing latex with an aqueous slurry of carbon black before coagulation. The slurry can be prepared

using a dispersing agent, but the dispersing agent interferes with curing and the stock is undercured unless extra accelerator is used. If the slurry is prepared without dispersing agent the dispersion of carbon black in the raw crumb is very poor, but the carbon black readily breaks down in the compounding to give excellent dispersion in the finished stock. Vulcanisates prepared from dispersant-free masterbatch are said to be equivalent to those of a good dry rubber mix. W.R.M.

#### Measurement of Polyethylene Oxidation

A. Rudin, H. P. Schreiber, and M. H. Waldman

*Ind. Eng. Chem.*, **53** (Feb 1961) 137-140

A method using differential thermal analysis which is fast and reproducible gives results agreeing with older methods and is readily modified to operate at the temperatures and pressures used in processing. It is applied to mixtures of polythene and antioxidant, to show the rapid increase of oxidation above 150°C. Some measurements are made to show the effect of Carbon Black on low-density polythene with and without antioxidant. P.B.S.

#### Ultraviolet Absorbers in Plastics

C. P. Vale and W. G. K. Taylor

*Chem. and Ind.*, No. 9 (4 Mar 1961) 268-272

The nature of solar radiation and the accelerated testing of the light stability of plastics are outlined, and the degradation of different plastics by sunlight is reviewed. The requirements of an ultraviolet absorber are discussed, and compounds used or suggested are described. Their application in plastics is briefly reviewed. W.R.M.

#### PATENTS

#### Removing the Waxy or Greasy Handle of Polyethylene Films

Bjorksten Research Laboratories

*USP* 2,934,457 (26 Apr 1960)

Treating polyethylene film with Cl under oxidising conditions, e.g. passing it through a bath of aqua regia, imparts a handle similar e.g. to that of vinyl films. C.O.C.

#### Improving the Printability of Polyethylene

Olin Mathieson Chemical Corp.

*USP* 2,935,418 (3 June 1953)

The adherence of e.g. printing ink to polyethylene is much improved by briefly exposing the polyethylene to high-frequency, high-voltage electric discharge. C.O.C.

#### Vinyl Polymers containing a Chemically Condensed Azoic Coupling Component

Chemstrand Corp. *BP* 864,292 (U.S.A. 10 Sept 1958)

An azoic coupling component is mixed with a monomer containing at least one >C=C< group and the mixture polymerised. The polymer may then be dyed at any stage of manufacture, e.g. after being formed into filaments or as moulding powder, by treatment with an azoic diazo component. C.O.C.

#### Incorporating Pigments in Polystyrene

Styrene Products

*BP* 864,799 (22 Oct 1958)

Insoluble colorants, particularly inorganic pigments, can be successfully incorporated in polystyrene if the colorant is added in the form of a colour masterbatch made from a low mol. wt. polystyrene. C.O.C.

Relation between Permanent Set and Covalent Cross-linking in Alkali-treated Wool (VI p. 264)

Tiled Effect on Linoleum or the like (X p. 270)

Coating a Backing with a Thermoplastic Composition (X p. 271)

## XIV—ANALYSIS; TESTING; APPARATUS

#### Biological Methods for Testing Mothproofing Agents

W. Frey

*Melliand Textilber.*, **42** (Jan 1961) 102-107

Samples of treated and untreated wool are exposed in suitable containers to attack by moth larvae for a fortnight under standardised conditions (temp., r.h., size of samples, number of larvae per sample, age and weight of larvae). Evaluation is done visually. Extent of damage, number of surviving larvae, growth during test, and other biological facts are noted. These observations are of special importance when testing new compounds. Five degrees of moth resistance are distinguished. Instructions for breeding larvae are given. W.M.

#### Infrared Absorption Study of the Relation between Viscosity and Intermolecular Hydrogen-bond Formation for Solutions of Phenolic Compounds in Oils. II—Solutions of Alkylphenol in Mineral Oil

M. M. Quraishi and S. H. Zaidi

*Pakistan J. Scientific and Industrial Res.*, **3** (Jan 1960) 31-35

The theoretical formula relating H-bond absorbance to the relative viscosity depression  $\left(\frac{\Delta \nu}{\nu}\right)$  (cf. *ibid.*, **2** (1959) 21), viz.,  $-\frac{\Delta \nu}{\nu} = K \frac{C}{100} \left(1 - \frac{a_{\nu}/C}{a_{\nu}/100}\right)$  is now verified for

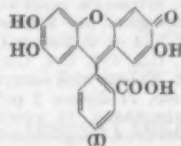
soln. of alkyl phenol, with  $K = 0.83 \pm 0.06$ , which compares well with  $0.77 \pm 0.04$  for *tert*-butylphenol,  $C_6H_4(OH)C(CH_3)_3$ , so that the mean  $(0.80 \pm 0.03)$  appears to be a generally applicable const. H.H.H.

#### Hydroxy-hydroquinonephthalein as a Metallochromic Indicator

V. Palaty

*Chem. and Ind.*, No. 7 (18 Feb 1961) 211

The title cpd. (I), prepared by a simple condensation of phthalic anhydride with hydroxyhydroquinone or with its more accessible triacetate, is used as an indicator in 0.2% ethanol soln. In acid soln. (pH 2-4) I is lemon-yellow with a weak green fluorescence and forms red complexes with the cations of Th, Zr, and Bi, at pH 5-7 it is orange-yellow and forms intensely purple complexes with Cu, Pb, Hg, and La, and in alkaline soln. (pH 8-10), it is pink, forming intensely purple-violet complexes with Ni, Co, Mn, Zn, Cd, and Mg. Ferric iron oxidises I and must be reduced by addition of hydroxylamine. In EDTA titrations, I is used in a similar way to Xylenol Orange; in acid media adjustment is made with 1-N HClO<sub>4</sub> or 1-N NaOH, hexamethylenetetramine is used for neutralising, and for titrations in alkaline media ammonia buffer soln. must be used. The complexing const. are low enough to permit indirect spectrophotometric determination of fluorides, phosphates, and sulphates, by measuring the decrease of absorbance of the Th and La complexes.



H.H.H.

#### Analysis of Mono-, Di-, Tri-, and Tetrakis-azo Aromatic Compounds

L. Sami

*Arch. Inst. Pasteur Tunis*, **33** (1960) 425-428

A review of general methods of qualitative and quantitative analysis.

#### Analysis of Monoazo Dyes

*Ibid.*, 429-441

The dyes are reduced with Sn and HCl, chromatographed, and the spots developed by spraying with an alcoholic solution of pyridine and contact with CNBr.

#### Reduction of Some Disazo Dyes. Qualitative Study

*Ibid.*, 442-449

The dyes were reduced with Sn and HCl, chromatographed and the spots developed by treatment with gaseous CNBr (König reaction). The specific spectra, *R<sub>f</sub>*, colour, and fluorescence are given. The principal dyes studied were Diazo Brilliant Yellow N, Sudan III (C.I. 26100), Bismarck Brown (C.I. 21000), Chrysophenine (C.I. 24895), and Congo Red (C.I. 22120).

#### Nitro Derivatives, Monoazo, and Disazo Compounds. Colouration of the Anils, and Fluorescence

*Ibid.*, 487-489

General review with four tables of the application of the König reaction. The fluorescence, colour, and variation of colour with the chromatographic method permit identification of the compounds.

#### Determination of Nitro Derivatives and Azo Dyes

*Ibid.*, 491-502

*Chem. Abs.*, **55** (6 Feb 1961) 2361

After the König reaction, areas of 1 $\mu$  of the nitro derivative or dye are evaluated photometrically. Tables are given which permit determination of the compounds examined. C.O.C.

### Chromatography and Ultraviolet Absorption Spectra of Aminoanthraquinones

Y. Bansho, I. Saito, and S. Suzuki

*Rept. Govt. Chem. Ind. Research Inst., Tokyo*,  
56 (Jan 1961) 12-20

Seventeen aminoanthraquinones and *N*-substituted-aminoanthraquinones were purified by adsorption chromatography on activated alumina. M.p. before and after this process are given.  $R_f$  values of these opd. were found by paper chromatography using four eluent systems; ethylene glycol-water, formic acid-glycerol-methanol, glycerol-methanol, and acetic acid-formic acid-water. The relation between ultraviolet absorption spectra, determined in alcoholic soln., and chemical structure is discussed. F.J.

### Quantitative Determination of Quinonoid Groups. Application to a Series of Quinones and to a Pyridine Extract of Coal

S. Delavarenne, A. Halleux, and H. Tschamler

*Rec. Trav. chim.*, 50 (Jan 1961) 27-42

Reductive acetylation with Zn and acetic anhydride or with Cu and  $H_2S$  followed by acetic anhydride is shown to furnish successful analytical methods for 30 model quinones. The Zn procedure failed, however, with a pyridine extract of vitrinite on account of oxide reactions with nitrogenous groupings and spurious hydrogenolysis of carbon-oxygen bonds. These difficulties were avoided in the Cu method, when an extract having a C content of 84.3% was found to contain 0.95% of quinone O, representing  $> 15\%$  of the total O. Since the sum of hydroxyl and quinone O represents 60% of the total O, the remainder is believed to be mainly in the form of ethers. H.H.H.

### Synthetic Dyes in Food and Drugs—Legislation, Toxicology, Manufacture, and Analysis

J. M. Plá Delfina

*Galencia Acta (Madrid)*, 12 (1959) 255-287

*Chem. Abs.*, 55 (6 Feb 1961) 2945

Review of current methods used in other countries, together with the author's own experience, for determining organic dyes in foods, cosmetics, and drugs. For the dyes permitted in Spain, extraction upon wool and with amyl alcohol and  $CHCl_3$  are given for: Yellow 1, 2, 3; Blue 1, 2; Orange 1; Red 1, 2, 3, 4, 5, 6; Yellow C-1; Blue C-1, C-2, C-3, C-4; Red C-1, C-2, C-3, C-4, C-5; Green C-1, C-2; Violet C-1, C-2, C-3. Colour with conc. and dil.  $H_2SO_4$  are given for 59 monoazo, 21 diazo, 2 polyazo, 1 diphenylmethane, 27 triarylmethane, 14 xanthene, 2 quinoline, 3 azine, 7 anthraquinone, 4 indigoid, and 1 phthalocyanine dyes. Colour reactions for aqueous and alcoholic solutions of dyes with conc. HCl and conc. NaOH are given for 31 monoazo, 4 diazo, 1 polyazo, 9 triarylmethane, 5 xanthene, 1 quinoline, and 1 indigoid dyes. C.O.C.

### Chromatography and Spectrophotometry of some Food Dyes

E. Miglietta

*Boll. lab. chim. provinciali (Bologna)*, 11 (1960) 216-229

*Chem. Abs.*, 55 (20 Feb 1961) 3983

The chromatographic values and spectrophotometric absorption curves of commercial samples of Ponceau 4R (C.I. Food Red 7), Ponceau 6R (C.I. Food Red 8), and Sunset Yellow FCF show many differences from those of the pure dyes. These differences depend on the source of manufacture and are presumably caused by impurities present in the commercial products. Only in the case of C.I. Food Yellow 4 did the  $R_f$  values and colour curves of pure and commercial samples agree. Chromatographic values with four eluents are given together with absorption curves. A standard method for giving chromatographic and spectrophotometric characteristics of food dyes is proposed. C.O.C.

### Paper Chromatography of Disperse Dyes

J. Gasparić and I. Táboriská

*J.S.D.C.*, 77 (Apr 1961) 160-161

### Characterisation of Sulphur Dyes

R. S. Chlenova, Ts. M. Gelfer, and L. V. Basova

*Zhur. priklad. khim.*, 33 (Feb 1960) 475-478

Sulphur dyes may be characterised with the aid of their absorption spectra (in the visible region of the spectrum) in dimethylformamide soln., comparison with the standard being effected by application of the Lambert-Beer Law. The method was tried successfully with a series of dyes obtained by aqueous polysulphide thionation of indo-anilines and derivatives of phenoxazines. T.Z.W.

### Conformation of Aqueous Diazo Compounds to Dilution Laws

B. V. Passet and B. A. Porai-Koshits

*Zhur. priklad. khim.*, 33 (Feb 1960) 496-499

Experimental evidence indicates that dilute aq. soln. of diazo compounds completely obey the laws of Bougher, Lambert, and Beer. T.Z.W.

### Methods of Testing Certain Mechanical Properties of Lacquer Films

F. M. P. Meredith and R. D. Guminaki

*J. Oil & Col. Chem. Assoc.*, 44 (Feb 1961) 111-118

The methods described were devised for the investigation of lacquer films applied to Al-Mg sheet. Certain properties are affected by the substrate, and tests on detached films are preferable. Detached films can be prepared either by applying the lacquer to lightly soaped sheet, from which they can be peeled, or, if the film-forming material is affected by moisture, the alloy sheet may be dissolved away by amalgamation with Hg. Methods for the determination of strength, extensibility, and elasticity of unsupported films are described. In determining adhesion, lacquer is applied to both sides of a panel which is then joined to platens by means of an adhesive; the lacquer film is pulled, through the platens, normally to the alloy surface. Scratch resistance is determined on the lacquered sheet with a conventional scratch tester, the needle of which is replaced by a hardened steel scraper. The scraper blade is  $\frac{1}{8}$ -in. wide, with an included angle of  $30^\circ$ , machined to a narrow flat to avoid digging into the metal, and is free to swivel on its own axis so that it aligns itself accurately whilst moving over the surface of the sample. There are several photographs and diagrams of the apparatus used in the tests. J.W.D.

### Potential Applications of Beta Gauges in the Textile Industry

O. Teszler, S. Ogawa, and H. A. Rutherford

*Amer. Dyestuff Rep.*, 50 (23 Jan 1961) 45-49

Experiments discussed in an attempt to show the usefulness of a beta gauge for the non-destructive measurement of weight (mass per unit area) include measurement of the wet pick-up, controlled continuous drying, fabric uniformity, and detection of fabric abrasion. S.B.D.

### Stripping of Procion Dyes from Cotton for Investigation of Faults

T. P. Nevell

*J.S.D.C.*, 77 (Apr 1961) 158-160

### Polarographic Determination of Tyrosine in Wool

L. Benisek

*Faserforsch. und Textiltech.*, 12 (Feb 1961) 74-78

The polarographic estimation of nitrated tyrosine is described. Determination of tyrosine in wool damaged by different treatments shows that the proportion of tyrosine in wool varies with the degree of damage by chlorine. W.R.M.

### Infrared Spectra of Polypeptides in Various Conformations: Amide I and II Bands

T. Miyazawa and E. R. Blout

*J. Amer. Chem. Soc.*, 83 (5 Feb 1961) 712-719

The amide I and II I.R. frequencies of the  $\alpha$ -helix, parallel- and antiparallel-chain pleated sheets and random conformations of polypeptides, including nylon, are explained in terms of vibrational interactions between adjacent peptide groups in the chain and through hydrogen bonds. Parallel and antiparallel pleated sheet conformations can be differentiated by frequency differences in the parallel amide I band, whereas the perpendicular amide I band and the parallel amide II band are common to both systems. Using these criteria many extended polypeptide chains (except  $\beta$ -keratin) were found to be in the antiparallel-chain pleated sheet conformation. I.R. bands of amide I and II were also obtained for random coil and  $\alpha$ -helix conformations, the directions of the amide I and II transition moments in the latter conformation of poly- $\gamma$ -benzyl-L-glutamate being inclined from the helix axis by  $29-34^\circ$  and  $75-77^\circ$ , respectively. F.J.

### Analysis of Fibre Mixtures. II—Binary Mixtures of Polyvinyl Chloride Fibres and Other Fibres

*Bull. Inst. Text. France*, (91) (Nov-Dec 1960) 109-112

Approved method, applicable to mixtures of polyvinyl chloride fibres with fibres of wool, silk, regenerated proteins, cellulose, polyamides, polyesters, polyacrylonitrile, polyvinyl alcohol or acetal, and glass. The polyvinyl

chloride is removed from a known dry weight of mixture by preferential solubility in an azeotropic mixture of carbon disulphide and acetone (55.5:44.5). J.C.F.

#### Improvement of Grey Scales for Determination of Colour Fastness (German Fastness Committee)

F. Gund *Melliand Textilber.*, 42 (Jan 1961) 85-86  
To avoid any possible differences between individual grey scales, every grey scale to be used for determination of colour fastness according to DIN 54000 series will in future be tested colorimetrically before being issued. W.M.

#### 203rd Meeting of the A.A.T.C.C. Technical Committee on Research

*Amer. Dyestuff Rep.*, 50 (20 Feb 1961) P142-P144  
Reports of current work of 10 research committees of the A.A.T.C.C. were presented, viz. RA63—Water Resistance of Fabrics, RA23—Colourfastness to Water, RA33—Colourfastness to Atmospheric Contaminants, RA34—Bleaching, RA49—Resistance to Insect Pests, RA60—Colourfastness to Washing, RA61—Wash and Wear, RA68—Determination of Odour in Resin-treated Fabrics, RA50—Colourfastness to Light, RA45—Identification of Finishes on Textiles. W.G.C.

#### Evaluation of Water-repellency

G. Mazingue, G. Messien, and M. Van Overbèke  
*Bull. Inst. Text. France*, (91) (Nov-Dec 1960) 27-43  
The water-repellent properties of samples of desized cotton fabric impregnated with 36 commercial preparations representing six principal classes of water-repellent compounds were investigated with the apparatus of Schwerber and that of Bundesmann. The two methods of test ranked the different classes in a different order, and the authors emphasise the importance, not only of standardising the individual test methods, but also of suiting the test method to the type of fabric and the requirements of the fabric in performance. The effect of washing and dry-cleaning on the treated samples was also investigated. J.C.F.

**Testing Resistance of Fabrics to Water-Absorption (T.T.S. No. 66)**  
Textile Institute *J. Textile Inst.*, 52 (Feb 1961) P79-P82  
A method for testing non-absorbent fabrics based on (a) inter-laboratory trials, (b) ASTM L1461-1956. Weighed specimens of conditioned fabric are immersed in distilled water at a constant temp. for a specified time. Surplus water is removed by mechanical shaking, the specimens are reweighed, and the water-absorption (%) is calculated. During immersion the specimens are constrained to lie in a horizontal plane at a given depth below the surface. J.C.F.

G. E. Settle *Ibid.*, P82-P86  
A summary of inter-laboratory and research work carried out in development of above test. M.T.

#### Acid Hydrolysis of Dimethylolurea Polymerised in Cotton

K. C. Eapen, J. R. Modi, and P. C. Mehta  
*Text. Research J.*, 31 (Feb 1961) 181-182  
Removal of the resin from the fibre depends not only on hydrolysis of the terminal  $N-CH_2O$ -Cellulose bonds but also of the  $N-CH_2N$  in the resin. Use of mild acidic hydrolysis enables distinction between the rupture of the two types of bonds. C.O.C.

#### Cuprammonium Insolubility as a Test for Covalent Cross-linking of Cellulose

R. D. Mehta and P. C. Mehta  
*Text. Research J.*, 31 (Feb 1961) 182-184  
Work showing that insolubility of resin-finished cellulose fibres is fallacious as an index of cross-linking. Treatment with 0.5-N NaOH at 20°C hydrolyses any cross-links formed between the  $N-CH_2OH$  of the resin and OH of cellulose. Similar hydrolysis occurs with the highly alkaline cuprammonium reagent. Thus in the case of cotton treated with dimethylol urea, insolubility of the treated resin in cuprammonium cannot be ascribed to presence of cross-links as they would have been already ruptured during the test. C.O.C.

#### Determination of Carboxyl Groups in Cellulose

O. Samuelson and B. Törnell  
*Svensk Papperstidning*, 64 (15 Mar 1961) 155-159 (in English)  
A new method which may be used to determine carboxyl

in the presence of carbonyl groups consists in the determination of the amount of acid necessary to transform the carboxyl groups from the sodium to the free acid form by elution with a known quantity of 0.01-N HCl. The method is useful for investigating alkaline degradation but cannot be used for commercial pulps. Results are in good agreement with those of Samuelson and Wenznerblom (*ibid.*, 58 (1955) 713) but much higher than those of other methods, presumably because carboxyl groups bound to lactones are not included in other determinations. 18 References. R.A.

#### Comparison of Kappa Number Test with other Methods for Determining Degree of Cooking of Chemical and Semi-chemical Pulps

J. Kaszyńska  
*Przegląd Papierniczy*, No. 10 (1959) 295-299  
*Polish Tech. Abs.*, No. 2 (38) (1960) 133-135

Experiments with unbleached sulphate, Kraft, and semi-chemical pulps using the Björkman, Silber, and Ostrand methods used in Poland and the Kappa method proposed by ICCA showed that the Kappa method gives more accurate results and there is a linear correlation between Kappa number and lignin content (up to 16% lignin). Conversion factors have been evaluated and the adoption of the method as a routine control test is suggested. R.A.

#### Testing Fastness of Coloured Leathers to Water Spotting

F. Jamieson  
*J. Soc. Leather Trades' Chem.*, 45 (Feb 1961) 51-54  
Development of the method given in the third report of the S.D.C. Fastness Tests Co-ordinating Committee (J.S.D.C., 71 (1955) 317). The following modifications are made—(a) no glass rod is used to rub the material; (b) colour change is assessed after drying overnight; (c) physical appearance of the leather after 30 min is recorded; and (d) a light manual treatment is included. J.W.D.

Coal-tar Dyes. XXIV—FD&C Red No. 4 (C.I. Food Red 1) (IV p. 259)

Study of Problems in Protein Chemistry of Non-hydrolytic Methods for Determination of Thiol and Disulphide (VI p. 264)

Interfacial Tension and Textiles (XV p. 276)

## XV—MISCELLANEOUS

#### The Eye, the Brain, and Land's Two-colour Projections

W. A. H. Rushton *Nature*, 189 (11 Feb 1961) 440-442  
Land has shown that the superposition of two black-white pictures, one through a red glass, can give rise to a coloured scene which includes greens and blues. The Land projection equipment, whereby red and white lights are superimposed in various proportions in 25 small squares (each subtending 1° at the eye), which together comprise the square projection field, has been used for several monocular and binocular matching experiments. Under monocular vision the squares gave a patchwork of many colours, and a three-colour (RGB) match was obtained with any square by projection from a Stiles double monochromator on to a white card, which was one-third the area of the square and between it and the observer. R, G, and B energies were measured by a photomultiplier tube. Binocular matches were obtained by observing the squares with the left eye, and the card (RGB) isolated by a tube with the right eye. It has been shown that the monocular matches follow Grassmann's law and the logic of three-cone pigments. Binocular matches show that the encoding of messages by the eye and decoding by the brain depend upon the surrounding field and its past history. E.COATES

#### Photoconduction in a Hindered *cis*-Isomer of $\beta$ -Carotene and its Relation to a Theory of the Visual Receptor Process

B. Rosenberg *J. Opt. Soc. Amer.*, 51 (Feb 1961) 238-240  
The temperature dependence of the dark current and photocurrent in 11,12:11',12'-di-*cis*- $\beta$ -carotene has been determined. The hindered *cis* isomer is  $10^6$  times more photoconductive than the all-*trans* isomer and  $10^3$  times more than the unhindered *cis* isomer. Rhodopsin is a

complex of the protein opsin with the hindered 11-*cis*-isomer of retinene; the significance of the results, as applied to the visual receptor process, is discussed.

E.COATES

#### Luminosity Functions of Normal, Deuteranomalous, and Deuteranopic Subjects as Determined by Absolute Threshold and CFF Measurements

W. F. Collins *J. Opt. Soc. Amer.*, 51 (Feb 1961) 202-206

The photopic luminosity relationships among one deuteranopic, two deuteranomalous, and two normal subjects have been determined by the absolute threshold and CFF (critical flicker frequency) techniques. CFF provides a very different kind of sensitivity measure from that of absolute threshold and visual acuity techniques.

E.COATES

#### Effect of Particle Separation on the Reflectance of Semi-Infinite Diffusers

W. R. Blevin and W. J. Brown

*J. Opt. Soc. Amer.*, 51 (Feb 1961) 129-134

Consideration has been given to the reflectance of pigments dispersed in a non-absorbing matrix. A special spectrophotometric arrangement was used and this consisted of a Beckman DU monochromator from which monochromatic light was directed on to the sample at an angle of incidence of 6°. The sample was held at the bottom of an integrating sphere of 1-m diameter. The total spectral reflectance was measured by comparison with an opal glass reference plate, by means of a photo-multiplier tube and potentiometric recorder. Investigations have been carried out on dry compressed pigments in addition to dispersions in water and turpentine. The results show that the reflectance of a semi-infinite diffuser with a non-absorbing matrix is independent of concentration over a wide range of concentration. Some attempt is made to explain deviations which occur at high pigment concentrations.

E.COATES

#### Caro and John Dale

M. Schofield

*Dyer*, 124 (2 Dec 1960) 845-846

Brief historical note.

M.T.

#### The Perkin Family

H. Wilkinson

*J.S.D.C.*, 77 (Apr 1961) 161

#### John Roebuck, F.R.S. (1718-94)—the First Sulphuric Acid Plant in Scotland

H. D. Turner

*Research*, 14 (Mar 1961) 118-120

A biography of the Yorkshireman who in 1749 established at Prestonpans the first sulphuric acid plant in Scotland. There was a large demand for sulphuric acid to replace the sour milk used in bleaching cotton. Before the works were erected the acid was being imported from England and Holland at 1s 4d per lb (previously the erection of Ward and White's works at Twickenham had brought the price down from 1s 6d to 2s 6d per oz to as much per lb) but by the end of 1776 the vitriol plant at Prestonpans was producing sulphuric acid for sale at 3½d per lb.

C.O.C.

#### Politics, Phoenix and Paterson "Textile Processing in New Jersey"

S. M. Edolstein

*Amer. Dyestuff Rep.*, 50 (20 Feb 1961) 123-128

Account of history of textile processing in New Jersey, U.S.A., particularly of the Phoenix Manufacturing Company of Paterson N.J.

W.G.C.

#### Education, Technology, and Textiles in Great Britain

D. B. Moore

*J. Textile Inst.*, 52 (Feb 1961) P63-P73

A survey, presented as a lecture at the Second International Week of Textile Technique in Barcelona on 7 Nov 1960. Discusses the British educational structure, technical education, the public-school system, education for the textile industry, the role of the Textile Institute, and the employment of technologists.

M.T.

#### Sulphur-containing Compounds—Nomenclature

F. O. Howitt *J. Textile Inst.*, 51 (Dec 1960) T651-T652

The present system of nomenclature used by the keratin chemist for relevant sulphur-containing compounds is somewhat ambiguous. A system has been evolved which is detailed for comment.

M.T.

#### Interfacial Tension and Textiles

G. Schwen

*Melliand Textilber.*, 42 (Feb 1961) 216-221

Relation between interfacial tension, contact angle, and capillary forces is discussed. Where high absorptive capacity is required (surgical dressings, handkerchiefs, towels, etc.) capillary forces must be strong. In textile printing these forces must be controlled to encourage penetration but prevent spreading. Capillary forces can have undesirable effects in pad dyeing, causing migration and unevenness. Importance of surface tension for wet processes and for water- and oil-repellent finishes is mentioned. Methods of measuring surface tension are discussed and a recording instrument based on Nouy's ring method is described.

W.M.

#### Process Control and Automation

T. J. Williams *Ind. Eng. Chem.*, 53 (Feb 1961) 166-167

A review of progress in 1960, with 110 references.

P.B.S.

#### Group Screening Designs

W. S. Connor *Ind. Eng. Chem.*, 53 (Feb 1961) 68A-69A

Describes the application of statistics to chemical processes, with the object of screening a large number of factors for their effect on the quality and quantity of the output. By the use of statistical designs of experiments the number of experimental runs is reduced.

P.B.S.

#### PATENTS

#### Colouring Coarse Aggregate

Minnesota Mining & Manufacturing Co.

*USP* 2,934,455 (2 Feb 1956)

Aggregate of screen size 0.25-4.0 in. is mixed with at least its own weight of finer aggregate and at least enough binder and colorant to give uniform coloration of the finer aggregate. The screen size of all the particles of the coarser aggregate must be at least double the weight-median screen size of the finer aggregate. After drying and firing the mass is separated into fractions one of which includes the coloured coarser aggregate. This is an economical method of obtaining a uniformly coloured product.

C.O.C.

#### Water-soluble Fluorescent-dye-coated Hydrophobic Silica

Monsanto Chemical Co.

*USP* 2,935,481 (20 Apr 1955)

Hydrophobic silica is treated with a fluorescent dye, e.g. Fluorescein (C.I. Acid Yellow 73), so that the dye is released only slowly when the silica is placed in or on water. The product is used e.g. as a visual marker on large bodies of water to point out the position of a particular object. A list of 31 suitable dyes is given.

C.O.C.

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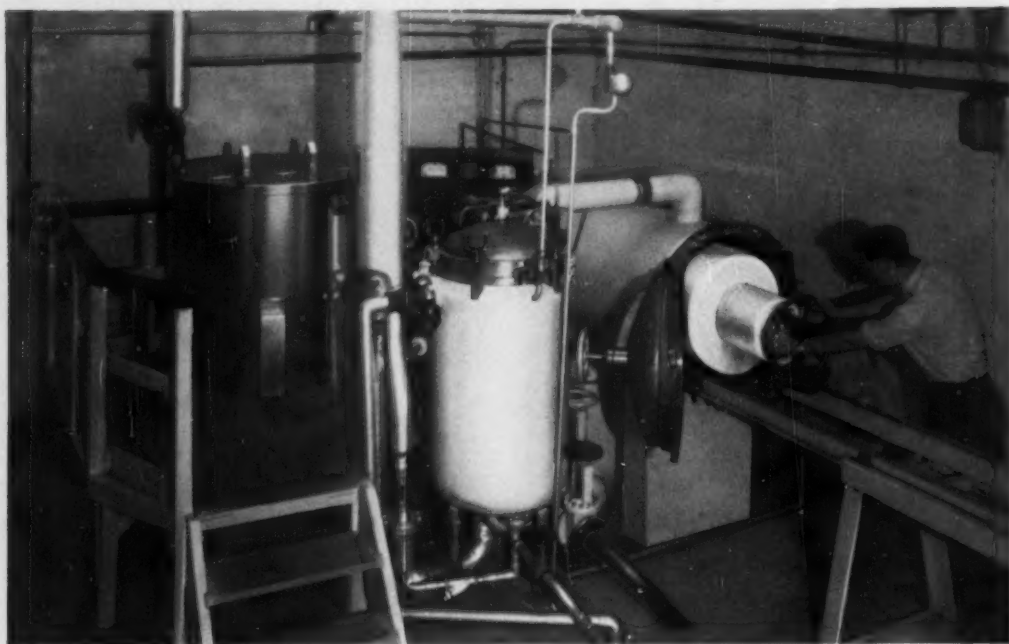
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THE Organisation's Division of Textile Industry, Geelong, Victoria, invites applications for research positions from Honours graduates in Science, or Engineering, with some years post-graduate research experience.

The Division, one of three constituting the Wool Research Laboratories, is broadly concerned with development of new physical and chemical techniques in wool textile processing and provides well equipped laboratories for fundamental studies and full scale mill equipment for application of results. The wide field of research include absorption studies on wool, fibre friction, permanent setting, shrinkage prevention, mechanical action of machines on wool fibres, servo-control mechanisms in processing and development of new machines.

The appointees will carry out duties in the following fields—

- (i) Chemical studies on modification of wool fibres. This work would involve the study of reactions with a view to developing new fabric finishes;
- (ii) The development of new physical methods for separating vegetable material from wool;
- (iii) Physical studies on fibre friction, and the effects of lubricants;
- (iv) Investigation of new methods of yarn production;
- (v) Fibre behaviour in processing using radioactive and high-speed photographic techniques.

Applications and enquiries from research workers in fields not strictly related to the above are also warmly invited.

Salary—Dependent on qualifications and experience within the ranges—£A1510-2265 p.a. or £A2395-2720 p.a.

##### EXPERIMENTAL OFFICER (No. 464/232)

THE Organisation's Division of Textile Industry, Geelong, Victoria, also invites applications for a position as Experimental Officer from graduates in Electrical Engineering, or in Science with Physics and Electronics as major subjects (or equivalent professional qualifications). Experience in design of electronic equipment is essential.

The appointee will be required to assume full responsibility for an electronics laboratory. The function of the laboratory will be to develop special electronic devices for application in research on woolen and worsted processing; to advise research staff in projects of an electrical engineering nature; and to provide servicing facilities for all electronic equipment of the Division. First-class laboratory facilities available.

Salary—Dependent on qualifications and experience within the ranges—£A1620-1950 p.a. or £A2070-2330 p.a.

Salary for a woman will be £A180 p.a. less than corresponding rates for men.

Promotion within C.S.I.R.O. is by merit and may ultimately go beyond the upper limit of the range within which the original appointment is made.

Fares paid. Further particulars supplied on application to—

Mr. E. J. Drake, Chief Scientific Liaison Officer, Australian Scientific Liaison Office, Africa House, Kingsway, London, W.C.2, to whom applications (quoting relevant appointment number) should be addressed by the 26th August 1961.

#### HINDLEY BROTHERS LIMITED HOLMEFIELD MILLS BARROWFORD NELSON

APPLICATIONS are invited for the position of—

##### DYE-HOUSE MANAGER

The man appointed must be capable of setting up and managing a new dye-house.

Qualifications in Chemistry and Dyeing essential, together with experience in pressure yarn dyeing techniques.

This is a staff appointment.

Full particulars, including age, and past experience to—The Managing Director, Hindley Brothers Limited, P.O. Box 20, Nelson Lancs. Telephone Nelson 62424-8.

A TEXTILE TECHNOLOGIST with wide experience of sizing and finishing processes is required to assist with the promotion of a new range of synthetic sizes and finishing agents designed specifically for use with the new man-made fibres. He should be able to give on-the-spot advice during mill trials. The salary offered will be commensurate with the experience required and the responsibility taken in this new and important position.

Schemes covering pensions, bonuses and other "fringe benefits" operate. Housing at reasonable rentals is available locally.

Apply Box 4086, c/o Charles Barker & Sons Limited, Gateway House, London E.C.4.

### TEXTILE TECHNOLOGISTS

IN DYEING AND FINISHING

BRITISH NYLON SPINNERS LTD. invite applications for a vacancy concerned, primarily, with development work on the dyeing and finishing of nylon knitted fabrics.

Applicants should—

- (1) Have had experience in the handling and processing of fine fabrics of man-made fibres, either as normal production or as experimental and development work.
- (2) Possess, preferably, a degree in Textile Chemistry or in Textile Technology. A lower academic level will be acceptable if it is combined with particularly wide and relevant practical experience.
- (3) Have the ability to co-operate with people of widely differing backgrounds.
- (4) Be between the ages of 27 to 37 years.

The works are situated in attractive country with easy access to towns. Rented houses are obtainable. Profit sharing and contributory Superannuation Schemes operate.

Applicants are requested to write, quoting T162, to the

Personnel Manager  
British Nylon Spinners Ltd.  
Pontypool, Monmouthshire

CHEMIST required by an old established firm situated on the borders of the Lake District. The business is concerned with the merchandising and manufacture of supplies for the laundry and dry cleaning industries. Knowledge of textiles, textile finishes, detergents, etc., required. The work includes development and evaluation of products for these industries and their introduction to laundries or dry cleaners. H.N.C. or equivalent qualification desired, superannuation scheme, applications giving details of age, experience and qualifications should be addressed to Box V 230.

EXPERIENCED WOOL SWEATER WASHER required for washing and fulling of knitted garments.

Applicants should have experience in the handling of Wool and Angora yarns or garments and should have been a washer or assistant head washer for a sweater manufacturer or a commercial finishing firm.

Salary within the range of £1300-£1500 p.a. with full company benefits, e.g. health insurance paid, sick leave, holiday payments, etc.

Applications to be addressed to—Mr. Robert Steubner, Personnel Director, Talbot Inc., Reading, Pennsylvania, U.S.A.

#### CALICO PRINTERS

wish to appoint a

#### PROCESS CHEMIST

to the Staff of their Machine and Screen Printing Works

THE person appointed will be initially responsible for quality control of cloth preparation, Ageing, Soaping and Finishing. The position offers ample scope for young technician possessing capacity and willingness to work hard.

Thorough practical experience in similar position considered more important than high academic qualifications, but H.N.C. essential. Salary will be commensurate with age and experience.

Applications, which will be treated in strictest confidence, should contain full details of experience, qualifications, and be addressed to—

Technical Director  
Stead, McAlpin & Co. Ltd.  
Cummersdale Print Works P.O. Box 1, Carlisle  
and endorsed "Chemist"

WE have a vacancy for a TOP DYER for wool and/or synthetic fibres. Only those having first-class qualifications and experience along with a good eye for colour should apply. This is an additional appointment, is permanent, and within a Staff Pension Scheme. Apply by letter giving full details—William Brook (Dyers) Ltd., Slaithwaite Dyeworks, Slaithwaite, near Huddersfield.

continued on page XXXVI

## APPOINTMENTS etc—continued

## APPOINTMENTS WANTED

**CHEMIST**, 13 years experience dyeing all textile fibres, also leather, requires position as chemist, dyer, or sales representative. City and Guilds. University educated. Box W 644

**DYESTUFFS TECHNICIAN** with five years experience in the field of Textile Screen Printing. Specialised capacity in the Printing of Pigments and other classes of Chemical Dyestuffs. Box W 647

**EXPERIENCED DYER**, aged 32, requires position. Eight years experience worsted, woollen and union cloths including Government contracts and indigo. Also loose materials. Box W 646

**MANAGER**, 27, full tech. cert., experienced cloth and pressure dyer, finisher and hand screen printer, seeks responsible progressive position. Abroad considered. Box W 645

**YARN DYER**. Cotton, wool and synthetic yarns. High temperature dyeing. Technical trained A.R.T.C. Salford and Final City and Guilds. Wide experience. Box W 643

## SCHOLARSHIPS

HUDDERSFIELD COLLEGE OF TECHNOLOGY  
WOOL TEXTILE RESEARCH COUNCIL  
SCHOLARSHIPS

## APPLICATIONS are invited for—

- (1) SCHOLARSHIP (annual value £350) for research in Wool Textiles.
- (2) SCHOLARSHIP (annual value £350) for research on Colour Vision.

The awards are tenable normally for a period of one to three years. Applicants, of British nationality, should have University degree or equivalent technological qualifications. Further details from the Principal.

H. GRAY

Clerk to the Governors



## TEXTILE REPRESENTATIVE MIDLANDS AREA

LAPORTE INDUSTRIES LIMITED seek a representative to sell their growing range of chemicals to the bleaching, dyeing and finishing sections of the Midlands textile industry. The selected person will have a background of practical experience of bleaching and finishing techniques, and he will, at the same time, be an above average salesman, able to negotiate at all levels of management.

He will have the personality and initiative to pioneer the sale of newly developed products for this specialised industry and at the same time will be responsible for maintaining the large existing turnover. He will have the backing of an excellent technical service organisation.

The selected person will receive a good salary, will be provided with a motor car, and will belong to the company's non-contributory pension scheme.

Applications should be addressed to The Group Personnel Manager, LAPORTE INDUSTRIES LIMITED, Hanover House, 14 Hanover Square, London, W.1, quoting reference LIL/JDC1/45.

### KAYSER BONDOR LIMITED BALDOCK

have a vacancy for a young man who has completed an apprenticeship in

#### HOSIERY and/or FABRIC DYEING and FINISHING

and has also had a sound theoretical training at an appropriate technical college

The position is a responsible one and offers excellent prospects. General conditions of employment, including a pension scheme, are of the best.

Applications should be addressed to the Senior Personnel Officer, Kayser Bondor Limited Baldock, Herts.

### MARKS & SPENCER

have a vacancy for a

#### TEXTILE TECHNOLOGIST

Applications are invited from men aged about 25/30 with training to A.T.I. or A.S.D.C. levels. Applicants should have practical experience in the dyeing and finishing of natural and man-made fibres, preferably in the Knitwear industry. The post is in the London Head Office but requires some travelling.

Please write giving age, qualifications, experience and present salary to the Staff Manager.

MARKS & SPENCER LTD.,  
MICHAEL HOUSE, 47 BAKER ST., LONDON, W.1.



## LIGHTNING FASTENERS LIMITED

*(A Subsidiary of Imperial Chemical Industries Limited)*

### A DYER

is required by Lightning Fasteners Limited, at WITTON, Birmingham.

The Company is setting up a modern dyehouse for the hank dyeing of zip fastener tapes, to commence operating in the early Autumn 1961. The successful applicant will take charge of the dyehouse.

Candidates should have several years' experience, a working knowledge of modern dyeing techniques and processes, and some supervisory experience.

Profit-sharing and Pension Fund schemes in operation. Assistance can be given towards house-purchase and removal expenses for a married man.

*Please write for an application form to the*

Personnel Manager (Staff)

IMPERIAL CHEMICAL INDUSTRIES LIMITED

Metals Division

P.O. Box 216 Kynoch Works

WITTON, BIRMINGHAM 6

**quoting LF/498/JSDC**

### TECHNICAL SERVICE

A man with initiative and a flair for developing new techniques, with experience in the chemical technology of textiles, is required to fill a senior post in the Technical Service Department of a large chemical manufacturing company.

The successful applicant will be responsible for the development of new and existing uses of the Company's products, which will involve visiting customers as well as supervising laboratory investigations.

The company offers a Pension Fund and Profit-sharing Scheme and a salary of up to £1750 to a suitable applicant. The vacancy is in the north-west of England. Apply to Box V229.

Telephone Bradford 682253-4 (Private Branch Exchange)

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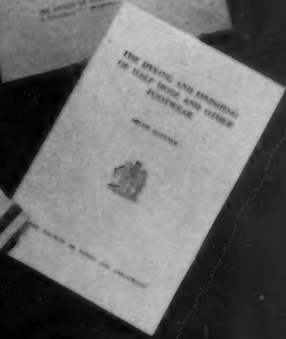
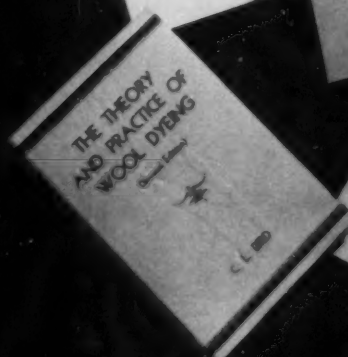
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